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## MATERIALS PROCESSING IN SPACE PROGRAM TASKS

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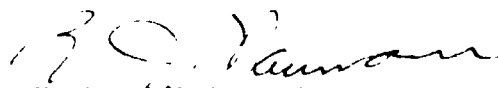
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*George C. Marshall Space Flight Center  
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16. ABSTRACT  <p>This report is a compilation of the active research tasks as of the end of fiscal year 1980 of the Materials Processing in Space Program, NASA Office of Space and Terrestrial Applications, involving several NASA Centers and other organizations. The purpose of this document is to provide an overview of the program scope for managers and scientists in industry, university, and government communities. The report is structured to include an introductory description of the program, its history, strategy and overall goal; identification of the organizational structures and people involved; and a description of each research task together with a list of recent publications.</p> <p>The tasks are grouped into six categories: Crystal Growth; Metals, Alloys, and Composites; Glasses, Ceramics, and Refractories; Fluids, Transport, and Chemical Processes; Containerless and Ultrahigh Vacuum Processes; and Bioprocessing. In many cases a task is placed in more than one category. For example, studies involving fluid dynamics of crystal growth were entered in both Crystal Growth and Fluids, Transport, and Chemical Processes. This insures complete coverage of each category.</p>					
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## NASA TECHNICAL MEMORANDUM

### I. INTRODUCTION

The Materials Processing in Space program is directed toward research in the science and technology of processing materials under conditions of low gravity to provide a detailed examination of the constraints imposed by gravitational forces on Earth. The program is expected to lead, ultimately, to the development of new materials and processes in commercial applications adding to this nation's technological base. The research studies emphasize the selected materials and processes that will best elucidate the limitations due to gravity and demonstrate the enhanced sensitivity of control of processes that may be provided by the weightless environment in space. Primary effort will be devoted to a comprehensive study of the specific areas of research which revealed potential value in the initial investigations of the previous decade. Examples of previous process research include growth of crystals and directional solidification of metals in the quiescent conditions in which gravitational fluid flow is eliminated, containerless processing of reactive materials to eliminate reactions with the container and to provide geometrical control of the product, synthesis and separation of biological materials in weightlessness to reduce heat and mass transfer problems associated with sedimentation and buoyancy effects, and identification of high vacuum characteristics associated with an orbiting wake shield.

Additional effort will be devoted to identifying the special requirements which drive the design of hardware to reduce the risk in future developments. Examples of current hardware studies are high-gradient furnaces and heat pipes to take maximum advantage of the lack of convection; acoustic, electromagnetic and electrostatic containerless processing modules; and electrophoresis separation devices.

In addition to the basic research nature of the program, a lower level of effort is being expended on the business, logistics and legal implication of rights of data and patents, control of materials, and division of responsibilities when NASA works with commercial ventures aimed at specific products. Examples of current materials research which might lead to commercialization include infrared detector crystals, inertial confinement fusion targets, electrolytes with dispersoids, aligned magnets, and ferromagnetic materials.

## History

Materials Processing in Space was initiated with a few simple demonstrations of principles by astronauts on Apollo missions 14 and 16. These were followed by more extensively planned but limited experiments on Skylab in 1974 which provided the first strong evidence of improved crystal growth, the elimination of convection, and other fluid effects in low gravity. On Apollo-Soyuz in 1975, many of the experiments were repeated with other materials and improved measurements. Also on that mission, electrophoresis experiments gave early indications of the possibility of improved separations of biological materials. In all, more than 50 flight experiments or demonstrations of fundamental effects of weightlessness were completed. Since the end of the Apollo program, the flight opportunities for researchers have been limited to ballistic rocket flights providing approximately 5 minutes of weightlessness during the coasting phase of flight. Approximately 30 experiments have been completed on Space Processing Applications Rockets (SPAR's).

A recent assessment of the program was made by a committee of the National Research Council on the Scientific and Technological Aspects of Materials Processing in Space (STAMPS). Recommendations stressed the need for more extensive ground-based research to serve as a support base for the evolution and assessment of investigations which would lead to a proper understanding of the role played by gravity in materials processes. Recourse to the weightless environment of space should be based primarily on the understanding and need in those specific cases identified from such a program. In addition, the first phase of the spaceflight program should be a demonstration of the new technology developed in the NASA program which should then be transferred to non-NASA entities for their use. The second phase, funded primarily by non-NASA users, should consist of a National Materials Laboratory in space to open the capabilities to all for a reasonable charge. Closer ties between the materials communities and NASA were recommended in the form of peer review of all proposals, both ground-based and spaceflight, and the periodic peer review of policies and plans. The Materials Processing in Space program has been restructured on the basis of these recommendations beginning with the earliest deliberations of the STAMPS Committee. An advisory committee has been formed to provide guidance in future program planning and policy making that is consistent with the spirit and principle of the STAMPS Committee recommendations.

### Program Strategy

The current program emphasis on fundamental processing science and technology in selected areas will continue for the period of this plan as the Materials Processing in Space program addresses problems of interest to the public and private commercial sectors which can be resolved by recourse to the space environment. During this phase of the program, the development and demonstration of current space technology for materials processing will be transferred, as appropriate, to non-NASA users. In order to assist this process, a Commercial Space Processing Task Team has been formed to resolve institutional constraints serving as disincentives to cooperative involvement. In addition, this team will serve as a single point of contact for interested parties and represent their interests within NASA.

Emphasis will be placed on the expansion of currently funded activities for ground-based and spaceflight investigations to maximize the outputs from these opportunities. Initiatives requiring new hardware will be encouraged at a low level until funds can be made available. The expansion of current efforts is occurring as a result of focusing support for current spaceflight investigations by forming facility experiment teams to provide advice and identify future involvement. At present, the two major facilities under development for Spacelab are the Fluids Experiment System, the Solidification Experiments System.

### Goal

The program demonstrates the capabilities of the space environment for materials processing to the scientific and commercial user communities and provides opportunities for independently funded users to exploit the space environment for processing related to their own needs.

### Objectives

The program has been structured to achieve the following specific objectives:

- Demonstrate the ability to achieve control over process variables at levels not achievable on Earth

- Control thermal fields
  - Increase compositional uniformity in crystals
  - Lower defect concentrations in crystals
  - Align internal structure of metal alloy systems
  - Increase purity of optical glass systems
  - Increase stability range of new glass systems
  - Increase geometrical uniformity of glass microshells
  - Reduce self-deformation and dislocation density of growing crystals
  - Improve effectiveness of electrokinetic separation
  - Produce large-diameter monodispersed polymer latex spheres
- Develop and demonstrate the capabilities of containerless processing techniques to handle and measure the properties of molten, reactive materials on which experiments cannot be performed in Earth-based laboratories
    - Viscosity
    - Equilibrium and dynamic vapor pressures
    - Enthalpy, emissivity, and temperature measurements
    - Phase equilibria studies
    - Enthalpy of solution and reaction
    - Processing of droplet arrays
  - Demonstrate the nature of the vacuum achievable in space and its utility for extending the range of important experimental parameters in extra-high vacuum science as well as its potential for achieving novel materials processing capabilities
  - Provide opportunities for independently funded scientific and commercial users to perform processing in the space environment

In addition to these activities, the program will also actively explore mission and hardware configurations which will reduce cost and provide sufficient power for experiments. These studies have already identified a free-flying mode of operation in combination with a power and support module which most effectively satisfies the program requirements. In the period 1980-84, these definition studies will identify a series of phase options to

provide such flight opportunities and then proceed with preliminary designs.

#### Low-Gravity Flight Projects

Low-gravity research is provided by several means to scientists to carry on experiments to verify ground-based predictions.

- a. Drop tube at Marshall Space Flight Center (MSFC) (containerless solidification with approximately 2 seconds of weightlessness).
- b. Drop tower at MSFC (approximately 4 seconds of free fall with a complete apparatus).
- c. KC-135 or F104 aircraft (up to 40 seconds of weightlessness in a Keplerian parabolic trajectory, available several times a year).
- d. SPAR (5 minutes of weightlessness in coasting flight after power cut-off; samples recovered after parachute descent); four more flights planned through 1980.
- e. Materials Experiment Assembly (MEA) (up to 5 days in orbit in the Space Shuttle. MEA is an autonomous package which will support five experiments with a minimum of integration effort).
- f. Spacelab materials processing experiments (5 or more days of weightlessness on the Space Shuttle); hands-on experiments operated by Mission or Payload Specialists in a laboratory environment. First flight planned for Spacelab 3.
- g. Materials Experiment Carrier (MEC) will be an automatic, free-flying satellite to support a large number of experiments for 3 or more months; it will use the 25 kW Power Module for support.

#### Format

The key to the format for listing Materials Processing in Space program tasks in this catalog is as follows:

- a. Title:
- b. Performing Organization

- c. Principal Investigator:
- d. Co-Investigator(s):
- e. Contract Number and Total Cost:
- f. Period of Performance:
- g. Abstract of Objectives, Approach and Results:
- h. Publications:

#### Program Organization

The NASA Materials Processing in Space program is administered by a Division Director and his staff in the Office of Space and Terrestrial Applications at NASA Headquarters, Washington, D.C. The Division Director is supported directly in the management of the program by the George C. Marshall Space Flight Center's Materials Processing in Space Project Office. That office depends upon the laboratories of the Marshall Space Flight Center, other NASA centers, other government agencies, universities, and industrial laboratories for technical support.



## **11. TASKS**

## A. CRYSTAL GROWTH

## Growth of Solid Solution Crystals

Marshall Space Flight Center

Mr. M. C. Davidson

Dr. L. R. Holland, Athens State College

Dr. A. F. Witt, MIT

Dr. D. D. Schenk, BMD-ATC

In-House Total Cost: \$540K (approx.)

October 1977 - October 1982

The major objective of this program is to determine the conditions under which single crystals of solid solutions can be grown from the melt in a Bridgman configuration. The central aim of this program is to assess the role of gravity in the growth process and to explore the possible advantages for growth in the absence of gravity. Since the HgTe rejected at the growth interface is more dense than the bulk melt, the system is stable against thermosolutal convection. However, the pressure of a dense component that lowers the melting point of the solid gives rise to a gravity-dependent interface shape instability. This is the primary focus of the experiment.

The problems of purity and containment in quartz ampoules have been resolved. The necessary purity and the resulting absence of chemical attack on the quartz was resolved by obtaining ultrapure starting material and loading by distillation. The structural integrity of the ampoules was studied. Crystals were grown by the Bridgman method and analyzed by the energy dispersive X-ray technique (Kevex). Composition was determined both longitudinally and radially. These compositional profiles are being analyzed by a one-dimensional and a two-dimensional model.

In addition to the basic studies, thermal profiles were determined to obtain the optimum growth environment for the HgCdTe material.

### Publications

Davidson, M. C. and Holland, L. R., "Narrow Zone Heating by a New Radiation Focusing Technique-Toroidal Ellipsoid Furnace," Rev. Sci. Instrum. 49, August 1978.

Davidson, M. C., Clayton, J. C., and Gillies, D. C., "Interface Shape Stability in Solid Solutions," J. Cryst. Growth, in press.

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Holland, L. R., "A Thermal Transmission Function for Fused Silica Ampoules," J. Cryst. Growth 49, 246 (1980).

Davidson, M. C. and Lehoczky, S. L., "Solidification of Solid Solutions - A One-Dimensional Analysis," submitted for publication in J. Cryst. Growth.

## Semiconductor Material Growth in Low-G Environment

Langley Research Center

Dr. R. K. Crouch

Dr. A. L. Fripp

In-Center Total Cost: \$260K (approx.)

February 1978 - February 1983

The principal purpose of this experiment is to utilize the microgravity environment of space to investigate the effect of convection on the homogeneity and perfection of compound semiconductor crystals. In a gravity field, the specific material  $Pb_{1-x}Sn_xTe$  being investigated has unstable solutal gradients or unstable thermal gradients depending on growth orientation.

Three different growth processes will be considered: (1) a vapor phase sublimation for seeded growth, (2) a modified Bridgman growth in which polycrystalline aggregate is necked down to encourage growth of a single crystal, and (3) a modified Bridgman melt back and regrowth.

Research in preparation for the space flight consists of both theoretical and experimental efforts. Numerical analysis of the mass and heat transfer will predict the furnace profile needed to obtain a planar isotherm at the melt-solid interface. Crystals grown on Earth will be used for comparison with those grown in space.

### Publications

Crouch, R. K., et al., "Growth of  $Pb_{1-x}Sn_xTe$ : Earth Options and Space Opportunities," 157th Electrochemical Society Meeting, St. Louis, Mo., May 1980.

Clark, I. O., Fripp, A. L., et al., "Diffusion Coefficients for Liquid  $Pb_{1-x}Sn_xTe$ ," 157th Electrochemical Society Meeting, St. Louis, Mo., May 1980.

Fripp, A. L. and Crouch, R. K., "Compositional Control Required in Alloy Semiconductors Used in High Performance Infrared Detector Arrays," Infrared Phys. 19, 701 (1979).

Crouch, R. K., "The Role of Thermodynamics in the Design, Optimization and Understanding of Semiconductor Growth in Space," 14th Thermophysics Conference, AIAA, June 1979.

Crouch, R. K., et al., "High Gradient Furnace Studies Using Modular Design," Gordon Conference on Crystal Growth, Santa Barbara, California, January 1979.

Fripp, A. L., et al., "Epitaxial  $\text{Pb}_{1-x}\text{Sn}_x\text{Te}$  on  $\text{Ba}_2$  Substrate," J. Electrochem. Soc. 124, 104C (1977).

## Crystal Growth of Device Quality GaAs in Space

Massachusetts Institute of Technology

Professor H. C. Gatos

Dr. Jacek Lagowski

NSG 7331      \$350K/year (approx.)

April 1, 1977 - continuing task

The experimental and theoretical efforts are aimed at the establishment of relationships among crystal growth parameters, materials properties, electronic properties and device applications of GaAs. Toward this goal steps have been undertaken for the development of new approaches to the preparation and characterization of GaAs. This extensive ground-based program constitutes a necessary step toward insuring successful processing of GaAs under zero-gravity conditions. Due to its unique scope combining crystal growth characterization and device-related properties and phenomena, this program bears directly upon exploitation of the potential of GaAs in device applications.

The research task includes the detailed study of the mechanisms of GaAs crystal growth from the melt and from solution and of the development of techniques for the characterization of materials and electronic properties on a microscale, e.g., composition, carrier concentration, mobility, diffusion length, and lifetime. Relationships between electronic properties and device performance are a part of the research task.

### Publications

Jastrzebski, L., Lagowski, J., et al., "Model of Liquid Phase Electroepitaxial Growth: GaAs," presented at Fourth American Conference on Crystal Growth, July 1978, Gaithersburg, Maryland.

Jastrzebski, L., Lagowski, J., et al., "Liquid-Phase Electroepitaxy: Growth Kinetics," J. Appl. Phys. 49, 5909 (1978).

Jastrzebski, L., Lagowski, J., et al., "Dopant Segregation in Liquid Phase Electroepitaxy: GaAs," presented at Fourth American Conference on Crystal Growth, July 1978, Gaithersburg, Maryland.

Jastrzebski, L., Lagowski, J., et al., "Determination of Carrier Concentration and Compensation Microprofiles in GaAs," submitted to J. Appl. Phys.

Lagowski, J., Walukiewicz, W., et al., "Derivative Surface Photovoltage Spectroscopy: A New Approach to the Study of Absorption in Semiconductors; GaAs," J. Appl. Phys. 50, 5059 (1979).

Slusarczyk, M. M. G., "Study of Electronic and Optical Properties of Gallium Arsenide Surfaces and Interfaces," Doctor's Thesis, MIT, 1979.

Kamieniecki, E., Lagowski, J., et al., "Wavelength Modulated Photo-Capacitance Spectroscopy," J. Appl. Phys., 1980, in press.

Kamieniecki, E., Kazior, T. E., et al., "A Study of GaAs-Native Oxide Interface States by Transient Capacitance," 7th Annual Conference on the Physics of Compound Semiconductor Interfaces, Estes Park, Colorado, January 1980.

Walukiewicz, W., Lagowski, J., et al., "Determination of Compensation Ratios in Semiconductors from Electron Mobility and Free Carrier Absorption: GaAs," 153rd Electrochemical Society Meeting, Seattle, Washington, 1978.

Walukiewicz, W., Lagowski, J., et al., "Electron Mobility and Free Carrier Absorption in GaAs: Determination of the Compensation Ratio," J. Appl. Phys. 50, 899 (1979).

Lagowski, J., "Microcharacterization of GaAs for Device Applications," Proc. 3rd Biennial Univer./Industry/Cov. Micro-Electronic Symp., May 1979, Lubbock, Texas, IEEE Conference Record, p. 1.

Gatos, H. C., et al., "Present Status of GaAs," NASA Contractor Report 3093, January 1979.

Jastrzebski, L., Lagowski, J., et al., "Outdiffusion of Recombination Centers from the Substrate into LPE Layers: GaAs," J. Electrochem. Soc. 126, 2231 (1979).

Jastrzebski, L., et al., "Effect of Growth Kinetics on Formation of Recombination Centers of GaAs," 155th Annual Meeting of Electrochemical Society, May 1979, Boston.



Bryskiewicz, T., Lagowski, J., et al., "Electroepitaxy of Multicomponent Systems," J. Appl. Phys., in press.

Imamura, Y., Jastrzebski, L., et al., "Defect Structure and Electronic Characteristics of GaAs Layers Grown by Electroepitaxy and Thermal LPE," J. Electrochem. Soc., 126, 1381 (1979).

### III-V Semiconductor Solid Solution Single Crystal Growth

Rockwell International

Dr. E. R. Gertner

Dr. M. D. Lind

JPL 955354 Total Cost: \$146K (approx.)

April 6, 1979 - December 6, 1980

The overall program task is to improve the quality of semiconductor substrate material used in epitaxial growth processes, since the quality of the epitaxial deposit is often limited by the quality of the substrate. To achieve the desired material improvement, the floating zone technique used in conjunction with a low-gravity environment will be used. This combination of technique and environment is capable of producing large, uniformly doped and compositionally homogeneous single crystals. The III-V solid solutions were selected for the initial growth experiments. Strain-free, homogeneous single crystals of III-V solid solutions have not been successfully synthesized due to the nature of their phase diagrams where the liquids and solids are of different composition. The  $\text{Ga}_{1-x}\text{In}_x\text{Sb}$  was chosen initially because it has the lowest melting point and vapor pressure of the III-V solid solutions. The research objective was to generate a compositionally homogeneous seed for future float zone experiments. However, technical difficulties with the original approach and recent advances in the vapor growth of III-V solid solution led to a reassessment of the initial approach and a redirection of the program to the single crystal growth of CdTe, a II-IV compound.

The research task will involve attempts to float zone small-diameter CdTe crystals because density, surface tension, and thermal conductivity considerations for CdTe preclude the use of float zoning for large crystals in normal gravity. Materials synthesized will be analyzed by optical microscopy, X-ray diffraction and topography, infrared spectroscopy, and electrical measurements.

## Epitaxial Growth of Single Crystal Films

Rockwell International  
Dr. M. D. Lind  
Dr. Roger L. Kroes, MSFC  
NAS8-31733 Total Cost: \$111K  
October 14, 1975 - April 30, 1980

The objective is to grow epitaxial films of gallium arsenide by liquid phase epitaxy (LPE) in low gravity and to compare them with films grown in normal gravity.

To investigate crystal growth in low gravity, an experiment was designed and performed on a Space Processing Applications Rocket (SPAR) flight. GaAs LPE appeared to be an appropriate process for investigation in such a flight because it can be completed within the few minutes of low gravity provided by the flight and because of broad interest in this process and material.

An LPE processor suitable for sounding rocket flights was designed and built. It incorporates a tubular resistance heated furnace, a system to provide a flowing H<sub>2</sub> atmosphere inside the furnace, a pneumatically operated slider, and a microprocessor for controlling the process. For a planned growth temperature of 720°C a molten solution of GaAs in Ga is saturated at 730°C and then, beginning at the launch, is lowered at a rate that allows the growth temperature to be reached within the first 4 minutes of the flight. By then the near zero-gravity condition is well established and the fluid motions induced by the launch are damped out. Growth is initiated at this time by sliding the substrates into contact with the solution and is terminated after 1 minute, but well before the end of the near zero-gravity portion of the flight, by retracting the substrates. With diffusion-limited growth, a film thickness of the order of 1 micron is expected.

### Publications

Lind, M. D., Kroes, R. L., et al., "Liquid Phase Epitaxy (LPE) of Gallium Arsenide in Low Gravity," 157th Electrochemical Society Meeting, St. Louis, Mo., May 11-16, 1980.

Lind, M. D., "Epitaxial Growth of Single Crystal Films," SPAR III Final Report, NASA TM-78137, January 1978.

Advanced Methods for Preparation and Characterization of  
Infrared-Detector Materials

McDonnell Douglas Research Laboratories

Dr. S. L. Lehoczky

Dr. F. R. Szofran

Dr. B. G. Martin

NAS8-33107 \$85K/year (approx.)

December 5, 1978 - December 4, 1981

The objectives of this research program are to quantitatively establish the characteristics of  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  as grown on Earth (1-g) as a basis for subsequent evaluation of the material processed in space and to develop experimental, theoretical, and analytical methods required for such evaluation.

A series of differential thermal-arrest (DTA) measurements were performed for  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  alloy compositions of  $x = 0, 0.1, 0.2, 0.3, 0.4, 0.6, 0.7, 0.8, 0.9, 1.0$ . The solidus and liquidus temperatures deduced from the DTA data were used to establish the pseudobinary  $\text{HgTe-CdTe}$  constitutional phase diagram and the  $x$  and interface temperature dependencies of the Cd segregation coefficient,  $k$ . Iterative phase-equilibria calculations, based on a regular associated solution (RAS) theory, were performed to establish the solution parameters required to calculate the phase boundaries for the  $\text{Hg-Cd-Te}$  alloy system. The calculated mercury partial pressures for the parameters are in reasonable agreement with published data.  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$  alloy crystals were grown by a high-temperature-gradient Bridgman method at furnace translation rates of 0.0685, 0.310, 0.597, 1.12, and 5.62 micron/sec. For three of the ingots, the longitudinal compositional profiles were measured and were compared with calculated profiles for the measured values of  $k$  and for various assumed values of  $D$ , the liquid  $\text{HgTe/CdTe}$  interdiffusion coefficient.

The mobility calculations performed thus far include the following intrinsic scattering mechanisms: longitudinal optical phonon, longitudinal and transverse-acoustical phonon, heavy-hole, and alloy disorder potential. The extrinsic scattering mechanisms include charged and neutral point defects. A comparison of calculated results with available experimental data indicates that longitudinal optical phonon and charged and neutral defect scattering are the dominant mobility-limiting mechanisms.

## Characterization of Semiconductor Materials

Marshall Space Flight Center  
Mr. M. C. Davidson  
Dr. D. C. Gillies, USRA  
In-House \$40K/year  
October 1979 - October 1980

The objective of this experiment is to develop the techniques for characterizing high-quality, solid-solution, alloy-type semiconductors for use as infrared detectors or as IR transparent substrates on which IR detectors can be grown by LPE or other techniques. Emphasis had been given to  $\text{Hg}_{0.8}\text{Cd}_{0.2}\text{Te}$  because of its importance as a detector material and because it is generally accepted as one of the most difficult materials of this type to grow on Earth. One of the major goals is to achieve a very low intrinsic carrier concentration in order to extend the bandwidth from the existing 2 GHz to approximately 5 GHz to satisfy a Department of Defense (DOD) requirement.

Chemical analysis data for melt growth experiments are necessary to test theories of crystal growth and obtain, indirectly, diffusion coefficients. In the case of the study of two-dimensional interface instability, these data may be necessary at high spatial resolution. An X-ray analysis technique using an energy dispersive system on a scanning electron microscope has been devised for determining  $x$  in the compound  $\text{Hg}_{1-x}\text{Cd}_x\text{Te}$ . Readings at spatial distances of 10 microns have been taken satisfactorily. With a minicomputer system now on line, the options exist for using a range of standards of different compositions and analyzing different spectral lines. These options are being actively pursued. Additional tasks being undertaken include: orientation determination and crystal perfection studies by a back reflection (LAUE) technique, and also metallographic studies for characterizing inclusions and grain boundaries.

## Marangoni Effect in Crystal Processing

Arthur D. Little  
Dr. Arthur D. Fowle  
Dr. M. C. Davidson, MSFC  
Dr. A. F. Witt, MIT  
NAS8-32946, Total Cost: \$552K (approx.)  
March 1978 - December 1980

The Spacelab experiment consists of measuring the freezing interface morphology and the velocity and temperature fields on the surface of a molten zone in a cylindrical sample of gallium-doped germanium in a crystal growing configuration. The nominal sample diameter is 0.7 cm, and the pulling rate is variable. Postflight characterization of the space-grown crystals will be made, and the results of the measured experimental variables will be compared with theory.

The experiment will be carried out under conditions in which molecular diffusion-controlled processes can dominate the behavior of the melt because gravity-induced convection is made negligible in the microgravity environment of Spacelab. This processing condition is desirable because the the scientific understanding and control of it would produce an immediate promise for producing better semiconductor, crystal products. However, flows driven by surface tension gradient (Marangoni effect) are predicted to upset the diffusion-controlled process ideal. A major purpose of the experiment, and the theory in support of it, is to examine the nature and importance of the Marangoni effect.

### Publications

Fowle, A. A., "Float Zone Experiments System (FZES) for Spacelab," AIAA 17th Aerospace Sciences Meeting, January 1979.

Fowle, A. A., et al., "Experiments on the Stability of Conical and Cylindrical Liquid Columns at Low Boiling Numbers," Third European Symposium on Material Sciences in Space, May 1979.

## Defect Chemistry and Characterization of (Hg, Cd) Te

Honeywell

Dr. H. R. Vydyanath

NAS8-33245 Total Cost: \$246K (approx.)

December 18, 1978 - December 17, 1981

( $\text{Hg}_{1-x}\text{Cd}_x$ )Te is a variable bandgap alloy semiconductor extensively used for infrared applications. Detector performance close to theoretical limits imposed by infrared imaging systems requires extreme control of compositional uniformity, carrier concentration, and carrier lifetime. The program deals with the study of the nature and concentration of the lattice defects incorporated into ( $\text{Hg}_{1-x}\text{Cd}_x$ )Te alloys as a function of the physiochemical conditions of preparation; namely, the temperature, the partial pressures of the constituent elements, and/or the concentration or the activity of the dopant being incorporated.

Undoped, donor-doped, and acceptor-doped ( $\text{Hg}_{1-x}\text{Cd}_x$ )Te samples are annealed at various temperatures in suitable Hg atmospheres. The samples are quenched to room temperature from the high temperatures. Hall effect and resistivity measurements are carried out at 77 K to determine the carrier concentrations and mobilities. The variation of the carrier concentrations as a function of the partial pressure of Hg and/or the dopant concentration is used to arrive at defect models for the doped and the undoped crystals. Once the defect models are established, the thermodynamic constants governing the incorporation of the defects are evaluated.

### Publications

Vydyanath, H. R., et al., "( $\text{Hg}_{1-x}\text{Cd}_x$ )Te Materials Studies," Proceedings of IRIS Detector Specialty and Imaging Group Meeting, Minneapolis, Minnesota, June 1979.

Vydyanath, H. R., "Defect Chemistry and Characterization of ( $\text{Hg}_{1-x}\text{Cd}_x$ )Te," J. Electrochem. Soc., 126 1979, p. 371.

Vydyanath, H. R., "Defect Studies in ( $\text{Hg}_{1-x}\text{Cd}_x$ )Te," in press.

Vydyanath, H. R., "Defect Structures of Indium Doped, Copper Doped, and Undoped ( $\text{Hg}_{1-x}\text{Cd}_x$ )Te," in preparation for submission to the J. Electrochem. Soc.

## Direct Observation of Interface Stability

Stanford University

Professor W. A. Tiller

Professor R. S. Fiegelson

Dr. Dennis Elwell

NAS8-33110 Total Cost: \$165K (approx.)

December 1, 1978 - October 31, 1980

This task will use a direct observation method to facilitate the careful test of interface stability criteria in a convective environment. This study will form the basis for future experiments under nonconvective conditions. The objective is a careful test of theory with experiment on a model system with all the significant material parameters being measured for this system. Such a careful test has not been performed heretofore.

The relevant parameters will be measured, including (1) diffusion coefficients of the solutes in the liquid, (2) phase diagram and effective distribution coefficients for the solute, (3) liquidus slopes for the chosen solutes, (4) thermal conductivities and diffusivities for both liquid and solid, (5) solid-liquid interfacial energy and its coefficient of anisotropy, and (6) a study of interface attachment kinetics as a function of orientation.

### Publications

Tiller, W. A., "Interface Field Effects on Solute Redistribution During Crystallization," J. Cryst. Growth, 1980.



## Solutal Convection During Directional Solidification

National Bureau of Standards  
Dr. S. R. Coriell  
H-27954B \$80K/year (approx.)  
April 1977 - continuing task

The objective of this task is to calculate and measure effects of convection caused by simultaneous temperature and concentration gradients on directional solidification, including determination of segregation effects in experiments done on Earth and estimation of the effect of microgravity and magnetic fields in avoiding such convection.

Theoretical methods are being developed to predict convective and interfacial instabilities during unidirectional solidification of binary alloys in the vertical direction. Linear hydrodynamic stability theory incorporating solidification boundary conditions has been formulated and has established some criteria for the onset of nonplanar solidification and nonuniform incorporation of solute in the resulting solid, including effects of magnetic fields. This research will delineate the conditions under which gravity-driven convection will occur during constant velocity unidirectional solidification.

### Publications

Boettinger, W. J., Coriell, S. R., et al., "NBS: Properties of Electronic Materials," NBSIR 78-1483, edited by J. R. Manning and R. L. Parker, 1978, pp. 15-68.

Boettinger, W. J., Coriell, S. R., et al., "NBS: Properties of Electronic Materials," NBSIR 79-1767, edited by J. R. Manning, 1979, pp. 25-81.

Coriell, S. R., Cordes, M. R., et al., "Convective and Interfacial Instabilities During Unidirectional Solidification of a Binary Alloy," J. Cryst. Growth, 49, May 1980, pp. 13-28.

## Vapor Growth of Alloy-Type Semiconductor Crystals

Rensselaer Polytechnic Institute  
Dr. Heribert Wiedemeier  
NAS8-32936 Total Cost: \$460K (approx.)  
March 1978 - March 1983

The objectives of this program are to investigate through systematic ground-based studies the effects of gravity-driven convection on the growth of single crystals of alloy-type semiconductors, to define optimum conditions for the growth of these materials in a microgravity environment, and to perform crystal growth studies in space. For this purpose, the systems  $\text{Hg}_{.8}\text{Cd}_{.2}\text{Te}$  and  $\text{CuInS}_2$  have been selected.

The ground-based research is concerned with the quantitative investigation of the chemical transport properties of the preceding materials in evacuated, closed ampoules of fused silica employing elemental iodine or metal-iodides as transport agents. The mass transport rates and crystal morphology of these systems are investigated as a function of total pressure for different temperature gradients and under horizontal and vertical stabilizing conditions. The inherent partial pressure and density gradients of the system and the presence of gravitational forces on Earth cause convective interferences with the transport and condensation process. Present results reveal that the surface morphology and chemical homogeneity of  $\text{Hg}_{.8}\text{Cd}_{.2}\text{Te}$  crystals obtained under vertical stabilizing conditions are improved relative to crystals grown under horizontal conditions. The crystal quality of  $\text{CuInS}_2$  shows similar improvements for the horizontal ampoule configuration with decreasing pressure (decreasing convective interference) of the system. The combined results of ground-based studies will lead to the definition of optimum growth conditions for the actual space experiments.

### Publications

Wiedemeier, H., "The Chemical Vapor Transport Properties of the  $\text{CuInS}_2$ -Iodine System," submitted for publication.

Wiedemeier, H., "The Chemical Vapor Transport Properties of the  $\text{Hg}_{.8}\text{Cd}_{.2}\text{Te}$ -Iodine System," submitted for publication.

Wiedemeier, H., "High Temperature Mass Spectrometric Studies and the Heat of Formation of  $\text{CuInS}_2$ ," submitted for publication.

Fluid Dynamics and Thermodynamics of Vapor Phase Crystal Growth

Rensselaer Polytechnic Institute  
Dr. Heribert Wiedemeier  
NAS8-33562 Total Cost: \$254K (approx.)  
January 1, 1979 - February 2, 1981

The primary objective of this program is to provide basic mass transport and crystal growth data which, combined with a thorough knowledge of the thermodynamics, will improve the fluid dynamic characterization of vapor transport systems.

The program is concerned with the investigation of the effect of systematic variations (1) of the relative importance of buoyancy-driven convection and (2) of diffusion and viscosity conditions on mass transport and crystal growth. These investigations will be performed in evacuated, closed ampoules for selected temperature gradients and partial pressures of the transport species. The specific experimental tasks include mass transport rate studies as a function of ampoule orientation, geometry, and the effects of inert gas additions. These will be correlated with crystal growth rate and morphology studies. In addition, the analysis of the vapor phase is an essential aspect of this project. The experimental efforts are supported by theoretical studies, including the thermodynamic and fluid dynamic characterization of the gas phase and the estimation of mass transport rates for different diffusive and diffusive-convective conditions. The materials investigated under this program include selected group IV elements and IV-VI compounds and halogens as transport agents. This choice is based on increasing complexity, known thermochemical and structural properties of these materials, and the existence of microgravity results for some of these systems investigated in previous Skylab and ASTP experiments.

Publications

Wiedemeier, H., "The Effect of Inert Gas on the Chemical Transport Properties of the GeSe-GeI<sub>4</sub> System," submitted for publication.

Wiedemeier, H., "The Effect of Orientation of the Density Gradient with Respect to the Gravity Vector on the Mass Transport Properties of the GeSe-GeI<sub>4</sub> System," submitted for publication.

Wiedemeier, H., "The Influence of Ampoule Geometry on the Mass Transport Properties of the GeSe-GeI<sub>4</sub> System," submitted for publication.

Wiedemeier, H., "The Effect of Homogeneous Chemical Reactions on the Mass Transport of Chemical Vapor Transport Systems," submitted for publication.

## HgI<sub>2</sub> Crystal Growth for Nuclear Detectors

EG&G, Inc.  
W. Schnepple  
Dr. L. Van den Berg  
H-34318B      Total Cost: \$646K (approx.)  
April 28, 1978 - April 28, 1983

The objectives of this program are to obtain a benchmark quality sample grown at low-g conditions and to study vapor growth phenomena under space conditions.

Ground-based crystals show a defect structure which impairs their performance as nuclear radiation detectors. These defects may be caused by the gravitational force acting on the crystal in its weakened state at the elevated growth temperature and by irregular convection patterns in the vapor during growth.

The program will be supported by ground-based research aimed at a more detailed understanding and description of the problems associated with the crystal growth process. Mechanical strength measurements have been performed (uniaxial compression tests) which show that the crystals exhibit slip parallel to the c-planes at stresses as low as 10 psi. Preliminary calculations using a simple linearized model indicate the oscillating instabilities in the convection part of the vapor transport system are unlikely, even at 1 g, provided that the utmost care is taken in the preparation of the crystal growth source material.

### Publications

Schieber, M., Schnepple, W. F., et al., "Vapor Growth of HgI<sub>2</sub> by Periodic Source or Crystal Temperature Oscillations," J. Cryst. Growth, 33, 1979, p. 125.

Schnepple, W. F., et al., "Growth of HgI<sub>2</sub> Single Crystals in Spacelab III," J. Spacecraft & Rockets, 16, 1979, p. 441.

## Fluid Dynamics of Crystallization from Vapors

University of Utah - Salt Lake City

Dr. F. Rosenberger

NSG-1534      Total Cost: \$160K (approx.)

June 1, 1978 - May 31, 1980

This program is aimed at obtaining fundamental insight into the complex physiochemical fluid dynamics of closed ampoule vapor crystal growth processes to the extent that a desired set of crystal growth conditions can be designed in advance. A more directly applied part of the program is concerned with the synthesis of ultrapure mercuric iodide and the vapor composition (stoichiometry) required for the growth of mercuric iodide high resolution radiation detector crystals.

A comparison of the analytical and numerical modeling possibilities and their relative merits with respect to accuracy and computation costs has been performed on a horizontal cavity with net diffusive-convective mass transport. The singular boundary conditions that arise in this situation were shown to be of little consequence for the accuracy of the concentration and total flux parameters of interest to crystal growth. As experimental prerequisite laser Doppler studies, an apparatus and seeding technique have been developed which allow for the determination of three-dimensional velocity fields in the range  $10^{-2}$  cm/s to 30 cm/s inside a closed cylinder in a closely controlled temperature gradient. Mass spectroscopic capability is being built up to unambiguously determine the actual composition of some of the "bench-mark" systems and to supplement efforts on the impurity analysis of technologically important solid state materials. Deviation from the "ideal" stoichiometry has been determined iodometrically with a resolution of  $10^{-3}$  mole fractions. These efforts supply the basis for planned vapor phase synthesis of  $\text{HgI}_2$  from the ultrapure elements and for the absolute calibration of ongoing thermogravimetric studies of the vapor-solid phase diagram of the  $\text{HgI}_{2+x}$  existence range.

### Publications

Rosenberger, F., "Fluid Dynamics in Crystal Growth from Vapors," J. Physchem. Hydrodynamics, 1, in press.

Markham, B. L. and Rosenberger, F., "Velocity and Concentration Distribution in a Stefan Diffusion Tube," submitted to Int. J. of Heat and Mass Transfer.

Markham, B. L., Greenwall, D. W., et al., "Numerical Modeling of Physical Vapor Transport in Vertical Ampoules," submitted to J. Crystal Growth.

Jhaveri, B. S. and Rosenberger, F., "On Singular Boundary Conditions in Diffusive-Convective Mass Transfer Across a Cavity," in preparation.

Vapor Phase of PbSnTe

Jet Propulsion Laboratory  
Dr. J. A. Zoutendyk  
NAS7-100      \$97K/year  
March 1, 1980 - March 1, 1981

This effort consists of various studies of the effects of gravity-driven convection on the growth of PbSnTe from the vapor as definition study for a possible flight experiment.



## UltraHigh-Vacuum Semiconductor Thin-Film Technology

Jet Propulsion Laboratory

Dr. F. J. Grunthaner

Dr. B. F. Lewis

Dr. J. Maserjian

In-Center \$80K/year (approx.)

July 7, 1978 - January 31, 1980

The objective of this program is to investigate ultrahigh-vacuum (UHV) semiconductor thin-film technology. The ultimate aim is the development of procedures for the production of cost-effective thin-film semiconductor solar cells. In order to achieve this objective, the process requirements and limitations of this UHV technology must be carefully examined in ground-based experiments.

The approach to semiconductor thin-film deposition emphasizes structural measurement at all stages of deposition or synthesis. It is essential to characterize all stages of growth and processing utilizing a full range of surface spectroscopies. Growth and analysis chambers must have a UHV interconnection to permit in situ measurements.

The deposition approach also emphasizes the synthesis of interfaces rather than simple material deposition. Growth by use of molecular beams in a UHV environment allows compositional control on a monolayer or even sub-monolayer scale. A monolayer or less of specific chemical species (or dopants) can have a profound effect on interface electronic properties. The desired abrupt compositional changes prohibit the use of high sample temperatures during or after deposition. Instead, nonthermal energy deposition by infrared, ultraviolet, and/or electron irradiation can induce desired chemical reactions and increased mobility at the growing surface.

The new techniques of in situ post-deposition laser annealing and grapho-epitaxial substrates have already been shown to produce excellent crystallization of amorphous films without degrading their compositional profiles.

## Ultravacuum Vapor Epitaxial Growth of Silicon

General Electric Company

Dr. C. A. Neugebauer

NAS8-33121 Total Cost: \$105K (approx.)

August 30, 1978 - March 31, 1980

The objective of this program is to develop an ultra-high vacuum deposition process for the growth of polycrystals of silicon onto a polycrystalline oriented metal substrate. An important objective is to establish vacuum and vacuum purity requirements for the process and possible rationale for extension of these experiments using the greatly improved vacuum conditions and pumping speeds available in a Space Vacuum Research Facility. In addition, experiment requirements for possible extension of the vacuum deposition work in a Space Vacuum Research Facility are to be documented. Support of the system definition design work for such a facility is to be provided.

Silicon films 5 to 10 microns thick were deposited onto 25 to 50 micron tungsten foils from a resistively heated silicon source. Vacuum levels in the low  $10^{-8}$  Pa range and substrate temperatures in the 450°C to 600°C range were employed. X-ray diffractometry, scanning and transmission electron microscopy, and metallographic examination of cross-sectioned and etched films were among the methods used to examine the deposited films.

It was concluded that oriented deposits can be obtained at substrate temperatures below 600°C. Two types of Si crystallites were distinguished: subgrains of ~1 micron or less diameter which were columnar and only slightly misaligned with respect to each other, and large parent grains subtending many subgrains and coinciding with the tungsten grains in the substrate. Increasing the gas pressure leads to more random deposits, indicating lower pressure might lead to more ordered growth.

### Publications

Frost, R. T., et al., "Ultra Vacuum Epitaxial Growth of Silicon," to be presented at 8th International Vacuum Congress, Cannes, France, September 22-26, 1980.

## Efficient Solar Cells by Space Processing

Ames Laboratory, Iowa State University  
F. A. Schmidt  
J. R. Williams, MSFC  
H-34329B      \$80K/year  
October 1, 1976 - December 14, 1979

The purpose of this work was to define a laboratory experiment that demonstrates the feasibility of preparing efficient Schottky barrier solar cells by space processing. The investigation was directed toward utilizing the non-contaminating space environment in the preparation of thin film silicon solar cells by epitaxial growth on various metal substrates. The resulting deposits were then characterized to determine which parameters are essential to enhance solar cell efficiency.

Thin films of electron beam evaporated silicon were deposited on molybdenum, tantalum, tungsten and molybdenum disilicide under ultrahigh vacuum conditions. Mass spectra from a quadrupole residual gas analyzer were used to determine the partial pressure of 13 residual gases during each processing step. Surface contamination and interdiffusion were monitored by in situ Auger electron spectrometry. The study produced three important results, namely: (1) metal silicide growth can be suppressed by use of a silicon source heavily doped with phosphorous, (2) the partial pressure of oxygen is influential upon the silicon grain size even at the  $10^{-10}$  Torr level, and (3) the first known preparation of silicon heteroepitaxy on a metallic substrate, namely  $\text{MoSi}_2$ , was achieved.

### Publications

Schmidt, F. A., Campisi, G. J., et al., "Efficient Solar Cells by Space Processing," Final Report, January 1980.

Schmidt, F. A., Shanks, H. R., et al., "The Preparation of Thin Film Solar Cells Under Very Low Pressure Conditions," Final Report, December 1977.

Bevolo, A. J., Schmidt, F. A., et al., "Polycrystalline Silicon on Tungsten Substrates," J. Vac. Sci. Technol., 16, 1979, p. 13.

## Solution Growth of Crystals in Zero-Gravity

Alabama A&M University

Dr. R. B. Lal

Dr. R. L. Kroes, MSFC

NAS8-32945 Total Cost: \$492K (approx.)

June 28, 1978 - June 28, 1983

In a low-g environment, buoyancy-driven convection effects in solution crystal growth are greatly reduced and, thus, one can study diffusion mass transport which in 1-g is masked by convective phenomena. Also, triglycine sulfate (TGS) crystals have technological importance for infrared detectors. The objectives of the experiment are (1) to grow TGS crystals from aqueous solution in low-gravity, (2) to investigate mass transport and heat flow in a diffusion-controlled growth system, and (3) to evaluate the feasibility, possible advantages, and technical potential of producing solution growth crystals in space.

Single crystals of TGS have been grown using conventional low-temperature solution growth method. Also, a unique technique of growing solution growth crystals by extracting heat at a programmed rate from the crystal through a semi-insulating sting has been developed. TGS crystals will be grown by this technique during the Spacelab 3 mission. Data on heat and mass transport in a diffusion-controlled system will be obtained using a laser holography technique. Analytical studies are under way to estimate growth rates in low-g conditions.

### Publications

Lal, R. B., "Solution Growth of Crystals in 0-Gravity," Semiannual Report, NAS8-32945, October 1978.

## Crystal Growth in a Spaceflight Environment

Jet Propulsion Laboratory

P. J. Shlichta

NAS7-100 \$128K/year (approx.)

October 1979 - October 1980

Experiments being developed for early Spacelab missions are directed at (1) determining the effect of residual and transient accelerations on the growth process and on resultant crystal perfection, (2) determining the effect of low-level forced convection, and (3) developing a better understanding of the crystallization process by measuring growth rates versus thermal and concentration gradients in the absence of convection.

Crystals will be grown from solutions at programmed cooling rates in controlled thermal geometries. Cameras will record time-lapse sequences of processed images of the experiment, e.g., by holography, schlieren, shadowgraphs, monochromatic absorption, interferometry, and/or reflected light. Analysis of these images will provide maps of the growth rate of the crystal and the temperature, concentration, and motion of the surrounding solution. These data will be compared with the distribution of defects and impurities in the crystal and with the accelerational history of the space flight. Results will provide guidelines for optimum experimental parameters for future Spacelab crystal growth experiments.

### Publications

Chen, P. S., Shlichta, P. J., et al., "Convection Phenomena During the Growth of Sodium Chlorate Crystal from Solution," J. Cryst. Growth, 47, 1979, pp. 43-60.

Chen, P. S., Shlichta, P. J., "Free Convection about a Rectangular Prismatic Crystal Growing from a Solution," Int. J. Heat and Mass Transfer, 22, 1979, pp. 1669-1679.

## Float Zone Experiments in Space

Ames Laboratory, Iowa State University  
Dr. J. D. Verhoeven  
H-34328B      \$82K/year  
October 1978 - October 1980

The objectives of the program were: (1) to determine if surface tension-driven convection in a float zone can be controlled or eliminated by means of surface films and (2) to investigate solute distribution and measure liquid diffusion coefficients in floating zones. If surface tension-driven convection can be effectively controlled or eliminated, it should be possible to obtain homogeneous solute distributions in space, which is not possible on Earth. Also, it should be possible to obtain accurate liquid diffusion coefficients for systems too reactive to be contained in capillary tubes.

Ground-based experimental work involves building an experimental float zone apparatus which will allow high vacuum capability plus control and measurement of temperature gradient, growth rate, and oxygen potential. Oxide layer thicknesses will be evaluated by depth profiling plus Auger analysis. For program task No. 1, experiments will consist of measuring the effect of oxide layer thicknesses upon interface shapes, and the radial and longitudinal composition profiles. For program task No. 2, solute profiles will be evaluated in the initial transient zones and the quenched zones.

## Analysis of the Float Zone Process

Massachusetts Institute of Technology  
Professor R. A. Brown  
NSG-7645/Supplement 1

This research effort involves a computational simulation of the fluid and heat transfer that takes place in a float zone crystal growth process. Analytical models are being developed for the melt-solid interface to examine the effects of both natural convection and Marangoni convection. Such models are essential for determining the conditions that must be controlled in order to obtain flat interfaces in either ground-based or spaceflight float zone crystal growth.

## Surface Tension-Driven Convection Phenomena

Case Western Reserve University  
Dr. Simon Ostrach  
Dr. J. Adin Mann  
NAS8-33015 \$226K/year (approx.)  
May 8, 1979 - May 8, 1980

Very little detailed information exists on the behavior of free-surface liquid configurations under non-isothermal and/or nonisoconcentration conditions. Because such situations are inherent to the containerless processing of materials, the present research program is directed to developing theoretical and experimental techniques to describe and define the nature of surface-tension gradient induced flows and the associated transport phenomena. Surface and bulk flows generated by temperature and concentration (excess) gradients and their coupling will be determined. In addition, extensive characterization of the surface properties will be performed as an essential input to the detailed studies mentioned previously. The development of new instrumental techniques for determining surface flows is an important aspect of this project.

Proper analysis of free-surface problems requires the determination of the surface shape and temperature distribution from the solution. Because of the complexities associated with these, problems of progressively increasing generality will be studied. In this way the efficacy of various approximate methods, such as variational methods and the method of weighted residuals, will be investigated to help reduce the time and cost required to obtain physically meaningful solutions. By a technique developed as part of the present research program surface-tension gradient flows can be studied in deep liquid layers. Flow structures over a range of parameters for various heating modes will be determined.

Experimental study will be made of the interaction of the two types of flows in a small liquid column supported between two rotating ends. Both co- and counter-rotation will be investigated. Laser light-scattering techniques that detect the ripplon Doppler shift are being used and developed to follow the flow fields at surfaces.



### **Publications**

**Pimputkar, S. M. and Ostrach, S., "Transient Thermocapillary Flow in Thin Liquid Layers," accepted for publication in Physics of Fluids, 1980.**

**Basu, S., "Thermally Induced Interfacial Fluctuations," M.S. Thesis, Case Western Reserve University, summer 1979. Three papers have been prepared from this work for submission during the next few months.**

## Surface Tensions and Their Variations with Temperature and Impurities

National Bureau of Standards  
S. C. Hardy  
H-27954B      \$190K/year (approx.)  
April 1977 - continuing task

The objective of this research is to obtain accurate measurements of surface tensions and their variations with temperature and impurities for gallium and other materials proposed for Marangoni effect experiments to provide data needed for design and analysis of these experiments.

The sessile drop technique is being used to measure the surface tension of liquid gallium as a function of temperature in vacuum and helium atmospheres. The measurements are difficult because gallium is reactive and readily contaminated by impurities which depress the surface tension. In addition, temperature variation of the surface tension appears to be unusually small. Thus, very high precision is required to measure this variation accurately. These measurements of surface tension are being combined with applications of modern surface analytic techniques, especially Auger analysis, to characterize the impurity content of liquid surfaces. This is important because surface tension values of gallium and many other liquids are very sensitive to the impurity concentration at the surface. The temperature dependence of the liquid surface tension of gallium in high vacuum was found to be linear between 150°C and 750°C with a slope  $-0.068 \text{ mJ/m}^2\text{°C}$ . At lower temperatures, significant curvature in the temperature dependence was found, apparently related to impurity effects.

### Publications

Hardy, S. C. and Coriell, S. R., "NBS: Properties of Electronic Materials," NBSIR 78-1483, edited by J. R. Manning and R. L. Parker, 1978, pp. 3-14.

Hardy, S. C. and Coriell, S. R., "NBS: Properties of Electronic Materials," NBSIR 79-1767, edited by J. R. Manning, 1979, pp. 3-24.

Thermocapillary Flows and Their Stability: Effects of  
Surface Layers and Contamination

Northwestern University

Dr. S. H. Davis

Dr. G. M. Homsy, Stanford University

NAS8-33881 Total Cost: \$400K (approx.)

June 1980 - June 1983

The proposed research concerns the theoretical analysis of the fluid mechanics and heat transfer of motions driven by surface-tension gradients. The object is an understanding of the convection accompanying the process of growing high-quality crystals in a  $\mu$ -g environment. The geometries considered include thin films, deep films and float-zone configurations. The particular aspects addressed are (1) the effects on steady Marangoni flow of contamination and the placement of third-phase films on the melt-gas interface, (2) the prediction of possible instabilities of Marangoni flows of pure melts and, (3) the effects on such instability criteria of contamination and surface films.

Calculation of flow and heat transfer characteristics is possible in the following limits: (1)  $A \rightarrow 0$ , (2)  $M \rightarrow 0$ , (3)  $C \rightarrow 0$ . These asymptotic limits, together with perturbation theory, may be used to analytically describe features of the structure of solutions to this class of problems. These analyses will be used to probe the effects of large classes of insoluble surface films on the steady flows generated by surface-tension gradients. It should be possible to describe what film characteristics are required to either suppress convection entirely, retard it to a given degree or even possibly augment it. The types of effects attributable to contamination would be determined. The magnitude of such effects could be calculated given experimental values for certain appropriate physical constants describing the material response of the contaminants.

Analytical Float Zone Experiments Systems (AFZES) - Phase B Study

Arthur D. Little  
Dr. Arthur A. Fowle  
NAS8-32946      Total Cost: \$771K (approx.)  
March 1978 - December 1980

The Analytical Float Zone Experiments System (AFZES) will provide the system and reusable hardware capability to perform a variety of basic experiments in the floating zone configuration for materials processing aboard upcoming Spacelab flights. A baseline preliminary design for the AFZES has resulted from this work.

Publications

Phase B Study, Analytical Float Zone Experiments System (AFZES), Volume I, Technical Summary Report, January 1980.

## **B. METALS, ALLOYS, AND COMPOSITES**

### Aligned Magnetic Composites

Grumman Aerospace Corporation

Dr. D. J. Larson, Jr.

NAS8-32948 Total Cost: \$652K (approx.)

July 1978 - July 1983

The objectives of this program are to contribute to understanding the role of convection on plane front solidification of eutectic and peritectic composites and the relationships between morphology and magnetic properties. In addition, assessment will be made of the commercial potential for processing binary composites in low-g.

The aim of this program is to evaluate the impact of convection (thermal and/or solutal) or coupled convective-diffusive transport on the plane front solidification of contained binary magnetic composites. Eutectic, off-eutectic, and peritectic solidification are under investigation. The low-g orbital environment will be utilized to study diffusion controlled solidification for experimental regimes that would be described as convective/diffusive regimes terrestrially. In addition, the relationships between solidification processing parameters, solidification microstructure, microchemistry, and magnetic properties are being studied.

### Publications

Larson, D. J., Pirich, R. G., et al., "Low-g Bridgman Growth of Eutectic MnBi/Bi Magnetic Composites," Fourth American Conference on Crystal Growth, Gaithersburg, Maryland, July 1978.

Pirich, R. G. and Larson, D. J., "Magnetic and Metallurgical Properties of Directionally Solidified Eutectic BiMn/Bi Composites," J. Appl. Phys. 50, 2425 (March 1979).

Pirich, R. G., Larson, D. J., et al., "High Rate Directional Solidification of BiMn/Bi Magnetic Composites," Conference on In-Situ Composites-III, Boston, Massachusetts, November 1978.

Larson, D. J., Pirich, R. G., et al., "Plane Front Directional Solidification of Eutectic Bi/MnBi," 17th Aerospace Sciences Meeting, New Orleans, Louisiana, January 1979.

Pirich, R. G., Larson, R. G., et al., "The Role of Processing Parameters on the Magnetic Properties of Directionally Solidified Bi/MnBi Composites," 17th Aerospace Sciences Meeting, New Orleans, Louisiana, January 1979.

Larson, D. J. and Pirich, R. G., "Bridgman-Stockbarger Growth of Bi/MnBi Eutectic Composites," AIChE Convention, Boston, Massachusetts, August 1979.

Pirich, R. G., Larson, D. J., et al., "Ground-Based SPAR and STS Studies of Plane-Front Solidification, Heat Transfer and the Magnetic Properties of Bi/MnBi Composites," 18th Aerospace Sciences Meeting, Pasadena, California, January 1980.

Larson, D. J., Pirich, R. G., et al., "Convective Influence of Off-Eutectic Plane Front Solidification of Bi/MnBi," to be submitted to J. Crystal Growth.

Pirich, R. G., Larson, D. J., et al., "Characterization of the Effects of Plane-Front Solidification and Heat Treatment on the Magnetic Properties of Bi/MnBi Composites," 18th INTERMAG Conference, Boston, Massachusetts, April 1980.

Sounding Rocket SPAR Experiments of the Magnetic and Microstructural Properties of Directionally Solidified Eutectic Bi/MnBi," 157th Electrochemical Society Meeting, St. Louis, Missouri, May 1980.

Larson, D. J., Pirich, R. G., et al., "The Influence of Partial Mixing on Directionally Solidified Bi/MnBi Magnetic Composites," Fifth Conference on Crystal Growth, Fallen Leaf, California, May 1980.

Ravishankar, P. S., "Unidirectional Solidification of Mn/Bi-Bi Alloys," Ph.D. Thesis, Clarkson College, August 1979.

Larson, D. J., "Zero-G Processing of Magnets (MA-070)," Apollo-Soyuz Test Project Summary Science Report, Volume I, NASA SP-412, 1977.

## Directional Solidification of Magnetic Composites

Grumman Aerospace Corporation

Dr. R. G. Pirich

NAS8-32219 Total Cost: \$217K (approx.)

February 1, 1977 - March 1981

Following the intriguing results obtained on the ASTP experiment, in which Mn-Bi eutectic directionally solidified in space was observed to have a finer microstructure and enhanced magnetic properties, extensive ground-based and flight investigation has been initiated. This has resulted in several significant findings.

The published phase diagram for Mn-Bi was found to be in error, and the composition used in the the ASTP experiment was, in fact, hyper-eutectic. The space-processed sample had finer microstructure than the control sample, whose composition varied because of convective stirring. The enhanced room temperature coercive strength was due to the heat treating that occurred in the gradient freeze solidification used in the ASTP furnaces.

The extremely high value for low-temperature coercivity shown by the ASTP samples does appear to be unique and significant. The magnetic performance is surely related to the microstructure, but attempts to duplicate the magnetic performance of this sample by ground processing have so far been unsuccessful. A eutectic sample processed on SPAR VI showed somewhat finer rod diameters compared to Earth-processed samples and also had finer spacing with more regularity. Magnetic data for this sample are not yet available.

### Publications

Pirich, R. G., "Space Processing of Permanent Magnetic Composite Materials," Annual Report, Francis Bitter National Magnet Laboratory, MIT, October 1978.

Pirich, R. G., "Comparison of Properties of Directionally Solidified Magnetic Compounds in Low-g and One-g Environments," COSPAR Meeting, Budapest, Hungary, June 1980.



Pirich, R. G., Larson, R. G., et al., "The Role of Processing Parameters on the Magnetic Properties of Directionally Solidified Bi/MnBi Composites," 17th Aerospace Sciences Meeting, New Orleans, Louisiana, January 1979.

Larson, D. J. and Pirich, R. G., "Bridgman-Stockbarger Growth of Bi/MnBi Eutectic Composites," AIChE Convention, Boston, Massachusetts, August 1979.

Pirich, R. G., Larson, D. J., et al., "Ground-Based SPAR and STS Studies of Plane-Front Solidification, Heat Transfer and the Magnetic Properties of Bi/MnBi Composites," 18th Aerospace Sciences Meeting, Pasadena, California, January 1980.

Larson, D. J., Pirich, R. G., et al., "Convective Influence of Off-Eutectic Plane Front Solidification of Bi/MnBi," to be submitted to J. Crystal Growth.

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Pirich, R. G. and Larson, D. J., "Magnetic and Metallurgical Properties of Directionally Solidified Eutectic BiMn/Bi Composites," J. Appl. Phys. 50, 2425 (1979).

### Ultimate Intrinsic Coercivity SmCo<sub>5</sub> Magnet

Charles Stark Draper Laboratory  
Dr. Dilip K. Das  
Dr. R. T. Frost, General Electric  
NAS8-33607      Total Cost: \$396K (approx.)  
September 24, 1979 - September 23, 1982

The objective of the proposed research is to produce Sm-Co magnets of reasonably high maximum energy product with intrinsic coercivity approaching the theoretical limit of 350 KOe. These magnets will have to be composed of extremely fine, defect-free crystallites. The goals will be accomplished by (1) preparation of highly pure Sm-Co alloy by levitation melting and solidification in oxygen-free vacuum, (2) comminution of the alloys to micron size particles in oxygen-free environment, (3) densification of the aligned alloy powder to near theoretical density without introducing contamination and preventing grain growth.

Even though permanent magnets fabricated from Sm-Co are the strongest known, their coercivities are in the neighborhood of only 30 KOe, a mere 10 percent of the theoretical potential. The reason for this low value has been determined to be the reversal of magnetization by the mechanism of domain nucleation (at comparatively small demagnetizing fields at defect sites) and motion of domain walls.

The main task will be to develop alloy comminution techniques for the production of fine powders in an oxygen-free environment and densification of the magnetically aligned powders to near theoretical density in oxygen and other contamination-free states with fine grain size.

## The Growth of Metastable Peritectic Compounds

Grumman Aerospace Corporation  
Dr. D. J. Larson, Jr.  
NAS8-32998 \$87K/year (approx.)  
October 1979 - October 1980

This is a ground-based effort to produce the metastable peritectic phase of  $\text{CoSm}_5$  by directional solidification. This research is a prelude to the extension of the work being done on Mn-Bi and high performance magnets, such as the Co/rare-earth systems.

Calculations on the size of the diffusion layer and the fluid dynamically stagnant layer ahead of the interface have been completed for both Sm-Co and Pb-Bi. Some directionally solidified ingots of Pb-Bi have been produced with differing thermal gradient-to-growth rate ratios. Not all of the phases have been identified yet, but some anomalous lines have been found that may indicate a metastable phase. A technique for directionally cooling Sm-Co alloys in an induction furnace has been developed, and some ingots have been produced with aligned dendrites.

The phases of Pb-Bi will be identified as a function of solidification variables and gravity vector. Solidification of Sm-Co will be carried out in a controlled directional furnace to compare the results with Pb-Bi. The magnetic properties of Sm-Co will be evaluated if it appears that there is potential for a high coercive force phase.

## Undercooling Studies in Metastable Peritectic Compounds

Marshall Space Flight Center  
Dr. L. L. Lacy  
In-House \$80K/year  
March 1, 1979 - March 1, 1982

The objective of this program task is to investigate undercooling and containerless solidification of metastable superconducting alloys  $Nb_3Ge$  and  $Nb_3Al$  and pure metal melts such as Nb; specifically, to investigate the structure and superconducting properties of undercooled Nb-Ge alloys and to determine the feasibility of forming metastable  $Nb_3Ge$  in bulk form.

Pure Nb droplets have been undercooled in excess of 500 K in free fall using the MSFC drop tube. The droplets form single crystals with no shrink cavity in the interior. The outer surface is rough, indicating the shrinkage associated with solidification was taken up by the interdendritic fluid.  $Nb_3Ge$  droplets have also been deeply undercooled and rapidly solidified in the drop tube. Unfortunately, accurate measurements of the amount of undercooling were not available at the time. An increase in transition temperature of  $\approx 2$  K was observed above the normal as-cast transition temperature (usually about 6 K), indicating that some of the A-15 structure may have been formed. New instrumentation has been added to the drop tube to obtain an accurate temperature history of the sample. The tests with the  $Nb_3Ge$  droplets will be repeated so that the superconducting performance can be related to the amount of undercooling.

### Publications

Lacy, L. L., Robinson, M. B., and Rathz, T., "Drop Tube Experiments on Undercooled Niobium and Niobium Alloy Droplets," 108th AIME, New Orleans, 1979.

Lacy, L. L., Robinson, M. B., "Containerless Undercooling and Solidification in Drop Tubes," submitted to J. Colloid and Interface Science.

Robinson, M. B., Rathz, T. J., et al., "Drop Tube Apparatus for Low-Gravity Solidification," NASA Tech Briefs, MFS-25242, 1980 (patent pending).

Niser, N. B., Lacy, L. L., and Robinson, M. B., "Containerless High-Temperature Calorimeter," NASA Tech Briefs, Spring 1979, p. 89 (patent pending).

Steinberg, J., Lord, A. E., Lacy, L. L., et al., "Production of Bulk Amorphous  $\text{Pd}_{77.5}\text{Si}_{16.5}\text{Cu}_6$  in a Continuous Low-Gravity Environment, accepted for publication in Applied Phys. Letters.

Rathz, T. R., "Superconducting Measurements on Nb-Ge Alloys Using Susceptibility Techniques," MS Thesis, University of Alabama in Huntsville, 1980.

## Liquid Phase Miscibility Gap Materials

S. H. Gelles Associates

Dr. S. H. Gelles

NAS8-32952 Total Cost: \$430K (approx.)

April 1978 - April 1983

The overall objective of the investigation is to determine the manner in which the microstructural features of liquid-phase miscibility gap alloys develop. The results of such a determination should make it possible to control the microstructures and the resultant properties of these alloys. The long-duration low gravity afforded by the Shuttle will allow experiments supporting this research to be conducted with minimal interference from buoyancy effects and gravitationally driven convection currents.

Ground-based studies on Al-In, Cu-Pb, and Bi-Ga alloys are presently being conducted to determine the effect of cooling rate and composition on the phase separation and solidification processes that influence the development of the microstructure in these alloys. Both isothermal and directional cooling experiments are being conducted. Some understanding of the influence of gravity will be deduced by conducting these ground-based experiments at different angles to the gravitational vector. The ground-based experiments will be used as a technological base from which flight experiments will be formulated and with which such flight experiments can be compared and judged. At the present time, isothermal and gradient freeze experiments in the Al-In system are contemplated for the Materials Experiment Assembly (MEA) to be flown on an early Shuttle flight. Isothermal, directional solidification, and containerless experiments are being planned for later Shuttle flights.

### Publications

Gelles, S. H. and Markworth, A. J., "Microgravity Studies in the Liquid Phase Immiscible System: Aluminum-Indium," AIAA Journal, 16, 431 (1978).

Gelles, S. H., Bhat, B. N., et al., "Phase Separation in Transparent Liquid-Liquid Miscibility Gap Systems," Workshop on NASA Fluids Experiments System, University of Huntsville, July 11-12, 1979.

Markworth, A. J., Gelles, S. H., et al., "Separation Kinetics of Immiscible Liquids in a Low-Gravity Environment," Nuclear Met. 20, 1023 (1978).

Gelles, S. H. and Markworth, A. J., Agglomeration of Immiscible Liquids, Experiment 74-30," SPAR V Final Report, NASA TM-78275, August 1980.

Directional Solidification of Monotectic and  
Hypermonotectic Aluminum-Indium Alloys under  $\mu$ -g

Centre d'Etudes Nucleaires de Grenoble  
Dr. C. Potard  
No Funds Exchanged  
September 1976 - present

The objective of this program is to analyze the mechanisms involved in the composite solid structure formation obtained from a miscibility gap alloy under microgravity. The metallic system aluminum-indium has been chosen for its low critical temperature, broad miscibility gap, and rather well-known thermodynamic properties.

Solidification of 10 and 50 atomic percent In samples will be carried out under a directional gradient in a SPAR rocket. Previous isothermal experiments by Gelles with this system resulted in almost complete phase separation in low-g with the In-rich material surrounding a core of Al-rich material. The mechanisms responsible for this separation are not understood, but because In preferentially wetted the alumina crucible, it is believed that capillarity may play an important role in the phase separation. The present experiment will use a SiC crucible which is preferentially wetted by the Al. The directional gradient will be used to investigate the effects of droplet migration resulting from differential variations of the interfacial energy with temperature. Four experiments are required to obtain sufficient information to sort out the various effects.

The supporting ground-based research consists of: (1) Al-In phase diagram re-determination in the experiment composition range, (2) wetting properties of the two-phase liquid against silicon-carbide, (3) Earth gravity orientation effect on solid structures, (4) preliminary observations on capillary forces and coalescence, and (5) liquid-liquid and liquid-solid phase transformation studies (undercooling, kinetics, volume change).

Publications

Potard, C., "Proposal for a Space Processing Rocket Experiment: Unidirectional Solidification of Monotectic and Hypermonotectic Aluminum-Indium Alloys, Internal Report, 1976.



Potard, C., "SPAR Experiment 76-51: Experiment Readiness Review," May 18-19, 1978.

Boissier, J., Dusserre, P., and Potard, C., "Tests de Vibration et Vibration-Choc des Cartouches Prototypes SPAR," Internal Report, 1978.

Potard, C., "Preliminary Thermal Tests of the SPAR 76-51 Cartridges," Internal Report, 1978.

Potard, C., "A Review of the Mechanisms Involved in the Directional Solidification of Al-In Emulsions at Zero-Gravity," J. Brit. Interplan. Soc., Vol. 31, 1978, pp. 275-280.

Potard, C., "Directional Solidification of Al-In Alloys in Micro-gravity: Results of Basic Preparatory Investigations," 17th Aerospace Sciences Meeting, New Orleans, Louisiana, January 15-17, 1979.

Potard, C., "Directional Solidification of Al-In Alloys at Micro-gravity: Results of Basic Preparatory Investigations," Third European Symposium, Grenoble, France, April 1979.

Potard, C., "Interet de Courtes Perodes d'Impesanteur pour L'Etude de la Solidification des Alliages Immiscibles Al-In," Proceedings of Esrange Symposium, Ajaccio, April 1978, ESA SP-135.

Potard, C., "Structures de Solidification des Alliages Immiscible Al-In et Al-Cd," 22nd Metallurgical Colloquium, Saclay, France, June 19-21, 1979.

## Studies of Model Immiscible Systems

Marshall Space Flight Center

Dr. L. L. Lacy

In-House \$50K/year

October 1, 1979 - October 1, 1982

The objectives of this program task are to use model organic immiscible systems to obtain fundamental information applicable to two-phase systems in general and to apply this understanding to materials of interest in the Materials Processing in Space program and to the interpretation of flight experiment results.

A number of model immiscible systems have been investigated. Two systems, diethylene glycol/ethyl salicylate and cyclohexane/methanol have been purified and characterized in terms of phase diagrams and surface activity. Measurement of droplet migration in thermal gradients has been initiated using small dimensional cells to suppress convective flows. Such measurements will also be conducted on KC-135 flights. Holographic techniques are being explored to give size distributions at different times in a volume large enough to avoid wall effects.

## Publications

Ross, S. and Nishioka, G., "Foams: Proceedings of Symposium Organized by the Society of Chemical Industry, September 1975," Academic Press, London, 1976.

Nishioka, G. M., Lacy, L. L., and Facemire, B. R., "Surface Activity in Binary Miscibility Gap Systems," submitted to J. of Colloid and Interfacial Science.

Lacy, L. L., Nishioka, G. M., and Ross, S., "Investigations on Transparent Liquid Miscibility Gap Systems," Proceedings of Fluids Experiment System (FES), UAH, July 1979.

## Comparative Alloy Solidification

Marshall Space Flight Center  
Dr. M. H. Johnston  
In-House      Cost: N/A

This effort is an extension of the earlier SPAR experiments, which used transparent model systems to investigate the gravitational influence on the solidification process, to actual metallic systems. Effects such as macro and micro segregation, grain size, shape, and orientation, and physical properties of ingots cast in low-g will be compared to identical castings in unit and high-g environments. A striking decrease in grain size with increasing g-field has already been demonstrated, confirming earlier predictions that dendrite multiplication was influenced by gravity-driven convective flows.

A casting furnace has been completed and flown on SPAR VII. This will be complemented by additional rapid-quench casting experiments carried out in KC-135 aircraft. A special furnace for this purpose has been constructed and is being tested for use beginning in mid CY 1980. Additional experiments could also be carried out in the drop tower when it is reactivated in mid CY 1980.

### Publications

Johnston, M. H. and Griner, C. S., "The Direct Observation of Dendrite Remelting and Macrosegregation in Casting," SPAR V Final Report, NASA TM-78275, August 1980.

## Directional Solidification of Monotectic Alloys

Marshall Space Flight Center  
Dr. M. H. Johnston  
In-House \$53K/year (approx.)  
October 1979 - October 1980

The objectives of this investigation are to study the effects of the growth parameters and the gravity influence on the directional solidification of miscibility gap materials. The importance of the effect of gravity vector and fluid flow on liquid segregation and interface stability will be determined by reversing the orientation of the furnace assembly and by processing the samples in the presence of a magnetic field.

Using Al-Pb as the base material, third elements will be selected to contribute to interface instability and to investigate three-phase immiscible solidification kinetics. By reversing the gravity vector and by processing the samples in a magnetic field, the individual effects of the growth parameters will be identified.

Some success has been obtained by using Fe dopants in the Al-Pb systems to break down the planar interface into a cellular interface. As the dense Pb phase nucleates and falls to the interface, it is trapped in the intercellular region and forms either fine rods or a series of fine droplets, depending on the composition, growth rate, and gradient.

### Publications

Parr, R. A. and Johnston, M. H., "Growth Parameters for Aligned Microstructures in Directionally Solidified Aluminum Bismuth Monotectic," Met. Trans. 9A, 1825 (1978).

The Influence of Gravity on the Solidification of  
Monotectic Alloys

University of Wisconsin - Milwaukee  
Dr. A. Hellawell  
NAS8-33727 Total Cost: \$115K (approx.)  
February 1980 - February 1983

The objective of this research is to examine the monotectic reaction using directional solidification methods in order to obtain aligned composite structures. One aspect of the problem is the separation of two liquids below a miscibility gap and their incorporation within a duplex growth front. Both surface tensions and relative densities influence this process, and the objective of this research program is to identify the gravitational influence.

The systems under examination include Al-In, Cu-Pb, Cd-Ga and a transparent analogue  $(\text{CH}_2\text{CN})_2\text{-H}_2\text{O}$  as well as the ternary systems Al-In-Sn, Cu-Pb-Al and Cd Ga-Al. The transparent analogue system will be examined in a temperature gradient stage on an optical microscope in order to study the detailed form of the duplex, solid + liquid growth front. The particular objective here will be to look at the incorporation or rejection of liquid droplets at the growth front.

Publications

Hellawell, A. and Grugel, R., Proceedings of Third Conference on In-Situ Composite, Boston, Massachusetts, 1978, pp. 39-48.

Grugel, R. and Hellawell, A., AIME Spring Meeting, Las Vegas, Nevada, 1980.

## Liquid Metal Diffusion in Solubility Gap Materials

Marvalaud, Inc.

Professor R. B. Pond

John M. Winter, Jr.

NAS8-33046 \$34K/year

April 27, 1978 - September 15, 1980

The ultimate objective of this program is to measure the diffusion rates of two liquid metals. The intermediate objective is to verify or disprove the investigators' suspicion that determining diffusion constants of solubility gap liquid metals in one "g" experiments will lead to erroneous results due to density-driven convection motion. If this is true, the determinations will have to be done in microgravity. The immediate objective of this program is to conduct ground-based determinations of diffusion constants of the molten lead-zinc couple (at temperatures above and below the consolute) using utmost ingenuity to sort out (or suppress) the effects of gravity.

A new diffusion geometry has been devised which permits establishing a sharp, oxide-free, and void-free interface between the diffusion couples at a well-defined time after the melts equilibrate at a selected temperature. After allowing appropriate intervals for interdiffusion, the couples are quenched and analyzed by methods of quantitative metallography, quantitative chemical analysis, and X-ray fluorescence. The experiment permits having "g" in any selected direction.

## Ultrapure Metals Preparation in Space

University of California - Los Angeles

Dr. R. F. Bunshah

Dr. W. A. Oran, MSFC

NAS8-33115 \$67K/year

August 7, 1978 - March 7, 1980

The objective of this program is to investigate the preparation of ultrapure metals in space by vacuum melting/distillation technique and to study the properties of ultrapurity metals (impurity content < 1 ppb).

The ultrapure high vacuum environment of an orbiting Molecular Wake Shield Facility or equivalent represents a unique environment for the preparation of ultrapure metals because it provides a very low pressure environment of contamination gas. Starting with vacuum-melted material, high-purity samples of chromium, vanadium, and molybdenum were prepared in a diffusion pump system by vacuum distillation at three different base pressures in the range of  $10^{-4}$  and  $10^{-6}$  Torr for each metal. Samples were analyzed for various metallic and nonmetallic impurity content. Decreasing pressure of the vacuum system resulted in lower non-metallic impurity content, as might be expected. Best results were observed for vanadium. While there were few discrepancies in the chemical analysis of metallic elements, the analysis showed that the impurity levels of these elements also decreased as the base pressure decreased.

### Publications

Agarwal, P., Bunshah, R. F., and Bevolo, A. L., "Ultrapure Metals Preparation in Space," Final Report, NAS8-33115, July 1980.

## Measurement of the Properties of Tungsten at High Temperatures

Rice University  
Dr. J. Margrave  
NAS8-33199 \$20K/year  
November 27, 1978 - June 1, 1980

This research is directed toward the measurement of the thermophysical properties of tungsten using containerless techniques. The properties of tungsten are of interest because it lies at the extreme of metal melting points and is a key datum in any extrapolation or interpolation process. In addition, difficulties in handling molten tungsten may establish the limitations of ground-based containerless systems in processing materials at high temperatures.

Samples are suspended containerlessly by an electromagnetic levitator. Additional heat is supplied by electron bombardment. Temperatures are measured by pyrometers. Heat capacities are determined from cooling curves, and total enthalpy is obtained using a drop calorimeter.



## Surface Tensions and Their Variations with Temperature and Impurities

National Bureau of Standards

S. C. Hardy

H-27954B      \$190K/year (approx.)

April 1977 - continuing task

The objective of this research is to obtain accurate measurements of surface tensions and their variations with temperature and impurities for gallium and other materials proposed for Marangoni effect experiments to provide data needed for design and analysis of these experiments.

The sessile drop technique is being used to measure the surface tension of liquid gallium as a function of temperature in vacuum and helium atmospheres. The measurements are difficult because gallium is reactive and readily contaminated by impurities which depress the surface tension. In addition, temperature variation of the surface tension appears to be unusually small. Thus, very high precision is required to measure this variation accurately. These measurements of surface tension are being combined with applications of modern surface analytic techniques, especially Auger analysis, to characterize the impurity content of liquid surfaces. This is important because surface tension values of gallium and many other liquids are very sensitive to the impurity concentration at the surface. The temperature dependence of the liquid surface tension of gallium in high vacuum was found to be linear between 150°C and 750°C with a slope  $-0.068 \text{ mJ/m}^2\text{°C}$ . At lower temperatures, significant curvature in the temperature dependence was found, apparently related to impurity effects.

### Publications

Hardy, S. C. and Coriell, S. R., "NBS: Properties of Electronic Materials," NBSIR 78-1483, edited by J. R. Manning and R. L. Parker, 1978, pp. 3-14.

Hardy, S. C. and Coriell, S. R., "NBS: Properties of Electronic Materials," NBSIR 79-1767, edited by J. R. Manning, 1979, pp. 3-24.

## Electrotransport of Solutes in Refractory Metals

Ames Laboratory, Iowa State University

F. A. Schmidt

H-34328B      \$20K/year

October 1, 1978 - December 14, 1979

The objective of this investigation was to further study electrotransport behavior of metallic solutes in refractory metals. This work complemented a previous project in which a prototype electrotransport experiment was developed for deployment in a molecular wake shield facility. Specific experiments were made in which the electric mobility, diffusivity, and effective valence of molybdenum, rhenium, tungsten and zirconium in thorium were determined in an effort to better understand the behavior of these solutes so that maximum efficiency of the electrotransport refining process can be achieved.

A space simulation chamber capable of achieving pressures of  $2 \times 10^{-12}$  Torr was designed, fabricated, and used to develop a prototype electrotransport experiment. Laboratory electronics for the experiment were also developed, and an automatic control system was used to heat the specimen. Complete assembly line drawings of the components of the experiment and specifications for a high-current power supply were developed. Composite rods of thorium metal having a sharp step in the concentration of the solutes of interest were resistively heated to various temperatures with a direct current in a low-density environment. The displacement of this interface was traced by laser ion mass spectrometry from which the velocity of transport, electric mobility, diffusivity and effective valence of the solutes were determined. All four solutes were found to migrate in the same direction as the electron flow. Rhenium and molybdenum were found to be very mobile, with tungsten somewhat slower. Zirconium was found to move at a rate near that of the self-diffusion of thorium, viz., approximately two orders of magnitude slower than rhenium and tungsten.

### Publications

Lunde, B. K. and Schmidt, F. A., "Light Sensor for Ultra-high Vacuum System Utilizing a Silicon Photovoltaic Cell," Appl. Optics, 15, 1976, p. 2625.

Lunde, B. K. and Schmidt, F. A., "Precision Sample-and-Hold Stores Voltage for a Week," Electronic, Vol. 49, 1976, p. 120.

Scherber, F. A., Schmidt, F. A., et al., "Pressure Dependence of the Fermi Surface of Th," Phys. Rev. B, Vol. 16, 1977, p. 4235.

Schmidt, F. A., Lunde, B. K., et al., "The Ultra Purification of Metals by Electrotransport Under Outer Space Environmental Conditions," Final Report, January 1977.

Schmidt, F. A., Outlaw, R. A., et al., "Electrotransport Purification of Thorium Under Low Pressure Conditions," J. Electrochem. Soc., Vol. 126, 1979, p. 1811.

Schmidt, F. A., Carlson, O. N., et al., "Electrotransport of Solutes in Refractory Metals," Final Report, January 1980.

Schmidt, F. A., Beck, M. S., et al., "Molecular Wake-Shield Precursor Experiment," Final Report, January 1979.

Crystal Nucleation in Glass-Forming Alloy and Pure Metal  
Melts Under Containerless and Vibrationless Conditions

Harvard University

Professor David Turnbull

NAS8-32691      Total Cost: \$120K (approx.)

June 23, 1978 - June 22, 1981

The main objective of this research is to characterize nucleation behavior in glass-forming alloy melts. Such experiments should indicate if formation of alloy glasses in bulk form is possible and, if so, what are the necessary conditions. The most favorable conditions would be those in containerless, vibrationless experiments in high vacua or inert atmospheres.

The crystal nucleation behavior of  $\text{Au}_4\text{Si}$  and  $\text{Pd}_4\text{Si}$  glass-forming melts and of pure Ni by the droplet technique is being investigated. It has been found that the onset undercooling,  $\Delta T_0$ , for copious nucleation in molten  $\text{Au}_4\text{Si}$  droplets varies widely with thermal treatments which alter the nature of the  $\text{SiO}_2$  film on the droplet surface. However,  $\Delta T_0$  as large as 1/3 of the liquidus temperature for some droplets was observed. Glass and crystallization temperatures of  $\text{Au}_4\text{Si}$ -based alloys are sharply increased ( $\sim 1^\circ$  per atom %) when Cu replaces some of the Au.

Publications

Turnbull, D., "On Anomalous Prefactors from Analysis of Nucleation Rates," NASA Quarterly Report, September 1979.

Spaepen, F. and Turnbull, D., "Negative Pressures and Melting Point Depression in Oxide-Coated Liquid Metal Droplets," Scripta Met., Vol. 13, 1979, p. 149.

Thompson, C. V. and Spaepen, F., "On the Approximation of the Free Energy Change on Crystallization," Acta Met., Vol. 27, 1979, p. 1855.

A Proposal to Measure the Viscosity of Molten Pd<sub>78</sub>Si<sub>16</sub>Cu<sub>6</sub> -  
A Critical Experiment to Support Possible Space Flights

Drexel University  
Dr. Arthur E. Lord  
NAS8-3372      \$29K/year (approx.)  
February 1979 - June 1980

Metallic glasses are materials that are cooled very rapidly to bypass crystallization. Due to the lack of crystallinity, these materials have very unique properties (very "soft" magnetic properties, high hardness, high yield stress, corrosion resistance, etc.) In general, only small sizes are available. To try and make larger samples the time-temperature transformation (TTT) curves must be determined in order to calculate the cooling rate ( $R_c$ ) needed for glass formation. The most important physical property needed for the TTT diagram calculation is the viscosity and its temperature dependence.

An oscillating crucible method for determining the viscosity of molten Pd<sub>78</sub>Si<sub>16</sub>Cu<sub>6</sub> alloy has been developed. This material is one of the easiest glass formers and hence can be made in bulk. The results for the auxiliary eutectic alloy Pd<sub>82</sub>Si<sub>18</sub> indicate an extremely high viscosity about 50 poise at the melting point. This is about 1000 times the value for other liquid metal systems. The viscosity of Pd<sub>78</sub>Si<sub>16</sub>Cu<sub>6</sub> is presently being determined, and with these data the calculations for the best TTT diagram viscosity will be obtained. Interesting results have also been obtained for the viscosity of Fe-Ni-P-B alloys.

#### Publications

Steinberg, J. and Lord, A. E., "The Viscosity of Molten Fe-Ni-P-B Alloys," J. Noncryst. Solids (submitted for publication).

Steinberg, J. and Lord, A. E., "The Viscosity of Molten Pd<sub>82</sub>Si<sub>18</sub> Alloy," J. Noncryst. Solids (submitted for publication).

Lord, A. E., Wouch, G., and Frost, R. T., "Preparation of Amorphous Ferromagnetic Materials through Containerless Solidification," SPAR IV Report, NASA TM-78235, January 1980.

Science Requirements Definition Study for the Electro-  
magnetic Containerless Processing Module (ECPM)

General Electric Company

Dr. R. T. Frost

Dr. W. A. Oran, MSFC

NAS8-33421      \$130K/year (approx.)

April 9, 1979 - May 31, 1980

The objective of this program is to define the science requirements for containerless processing experiments and the facilities required to carry out these experiments. Facilities considered should include a ground-based facility, extensions possible for high-temperature thermophysical property measurements using a KC-135-mounted facility and a Shuttle-borne ECPM.

The collection and analysis of science requirements is carried out through personal liaison with investigators active in topics related to containerless processing and through periodic workshops. The individual experiments requirements are then translated into requirements for experiment system components capable of fulfilling most experiment requirements. The work includes a conceptual definition for both an airplane-borne and a Shuttle-borne experiment facility.

Publications

Minutes of the Electromagnetic Containerless Processing Task Team Meetings, Valley Forge Space Center, April 20, 1979, and Marshall Space Flight Center, October 16-17, 1979.

## Containerless Processing Technology

NASA Marshall Space Flight Center

Dr. W. A. Oran

L. H. Berge

B. B. Ross

Dr. J. E. Rush, UAH

In-House Cost: N/A

The objective of this program is to develop the research area of containerless processing in a microgravity environment, including: (1) the organization of working groups of experts and potential users to help develop a ground-based research program and define possible flight hardware, (2) the development and, in part, definition of the limitations of various ground-based containerless processing techniques, and (3) the conduction of precursory experiments in these ground-based assemblies.

Single-axis acoustic levitation devices were utilized as a basis for developing a system to containerlessly process nonconducting materials. However, there are apparent difficulties in operating one of these devices in a ground-based processing system. If one attempts to spot heat a levitated specimen to a high temperature in a relatively cold gaseous medium, instabilities are apparently established in the acoustic standing wave field which result in the sample being ejected from the levitation well. Acoustic streaming influences the temperature of a specimen levitated in an "isothermal" furnace. The streaming pumps cooler air into the levitation region in a manner such that the temperature of the specimen appears influenced more by the transducer temperature than by the wall temperature.

A study of the use of airjet levitation devices to process nonconducting materials has begun. Airjet systems have been used to containerlessly process metals, although the EM generator was used for heating and the resulting field greatly helped to stabilize the metal when molten.

### Publications

Oran, W. A., "Proceedings of the Workshop on an Electromagnetic Positioning System in Space," NASA CP-2069, October 1978.

Oran, W. A., "Preliminary Characterization of a One-Axis Acoustic System," NASA TM-78213, January 1979.

Oran, W. A., "A Parametric Study of an Acoustic Levitation System," Rev. Sci. Instrum. 51, 626 (1980).

Oran, W. A., Some Limitations in Processing Materials in Acoustic Levitation Devices," IEEE Transactions on Ultrasonics, September 1979, pp. 482-486.

Oran, W. A., "Containerless Melting and Solidification, Proceedings of Electrochemical Society Meeting, St. Louis, May 1980.

Oran, W. A., "Containerless Processing of Metals with an Electromagnetic Levitation System," in preparation.



Thermodynamic Properties of Refractory Inorganic Materials  
at High Temperatures

National Bureau of Standards

J. H. Colwell

H-27954B      \$50K/year (approx.)

April 1978 - continuing task

The objective of this research is to develop containerless techniques for determining thermophysical properties of nonmetallic samples at high temperatures.

Two approaches have been considered for carrying out the low-gravity experiments. The first is analogous to a drop-calorimetry experiment in that the sample would be heated to a predetermined temperature whereupon its enthalpy would be determined by engulfing the sample in a calorimeter. This approach would be tedious since a single point would be obtained for each sample, and many points would be needed for the requisite data. The second approach, which is presently being pursued, involves heating a sample to a high temperature and then allowing it to freely cool while simultaneously measuring its surface temperature and total radiant energy loss as a function of time. Temperature gradients within the samples will complicate the interpretation of the experiments but will also permit thermal conductivities of the samples to be determined under certain conditions. The limiting parameters for these measurements are being determined by computer modelling experiments. Pyroelectric detectors are being tested for potential use in the measurement of the total radiation from the samples.

Publications

Colwell, J. H., "NBS: Properties of Electronic Materials," NBSIR 79-1767, edited by J. R. Manning, 1979, pp. 103-131.

## Uniform Dispersions by Crystallization Processing

Massachusetts Institute of Technology

Professor D. R. Uhlmann

NAS8-31350 Total Cost: \$398K (approx.)

July 1, 1975 - June 1, 1980

The program was concerned broadly with developing improved understanding of the interaction between second-phase particles and an advancing solidification front and with developing criteria for the solidification processing of two-phase composite materials in space.

In the experimental portion of the work, the critical velocity (i.e., the maximum growth rate for which a particle is pushed by a growing crystallization front) was measured for a variety of particles, ranging in size between 1 and 200 microns in both high and low entropy of fusion ( $\Delta S_f$ ) organic materials. The critical velocity was inversely proportional to melt viscosity, was lower in small  $\Delta S_f$  materials than in high  $\Delta S_f$  materials having the same viscosity, and decreased with increasing particle size. Space experiments were conducted to eliminate the possible frictional effects of the particles against the container walls. These experiments indicated that particle pushing by a crystal-liquid interface does occur under microgravity conditions and that particle pushing in microgravity occurs with characteristics similar to those found in 1-g.

### Publications

Uhlmann, D. R., Aubourg, P. A., et al., "Multiphase Dispersions by Crystallization Processing," COSPAR Space Research, Vol. XIX, Pergamon Press, 1979, pp. 525-529.

Uhlmann, D. R., "Behavior of Second-Phase Particles at Solidification Front," SPAR V Final Report, NASA TM-78275, August 1980.

Solid Electrolytes Containing Dispersed Particles: The Effect of a Dispersed Second Phase on the Ionic Conductivity of Solid State Electrolytes

Arizona State University - Tempe  
Dr. J. Bruce Wagner, Jr.  
NAS8-32937 Total Cost: \$358K (approx.)  
February 1978 - October 15, 1980

The purpose of the proposed experiments is to study the role of a dispersed second phase on the ionic conductivity of materials used as solid state electrolytes.

A second-phase dispersion may be obtained in several different ways. For example, a supersaturated solution may be heated so as to cause precipitation of the second phase. This process suffers from the fact that the precipitation generally begins at preferred sites (not uniform) and, furthermore, the precipitate changes size (grows larger) with time. On the other hand, a mechanical mixture of the dispersoid and matrix can be pressed into pellets either at low temperature (cold pressing) or elevated temperatures (hot pressing). The advantage of this method is that the pellets can be produced rapidly but only one at a time. Alternatively, the mechanical mixture can be melted and cooled. In this method, there is gravitational segregation, but many samples can be sectioned from one melted and solidified boule.

We have studied the effects of a second phase of an insulator randomly dispersed in an ionic conductor. In all cases studies using fine particles ( $\leq 1$  micron), an increase in ionic conductivity is observed with the maximum enhancement being  $10^3$  in the  $\text{AgI}(\text{Al}_2\text{O}_3)$  system. Moreover, we have developed the highest silver ion conducting electrolytes as yet reported. The significance of these results is that electrolytes can operate more efficiently at lower temperatures than heretofore found.

Publications

Chang, M. R. W., "Studies on Cuprous Chloride Containing a Dispersed Second Phase of Alumina Particles," M.S. Thesis, Arizona State University, December 1978.

Dubec, P. M., "Electrical Conductivity Studies on Undoped, Cadmium-Doped and Sulfur-Doped CuBr and on CuBr Containing  $\text{Al}_2\text{O}_3$  Particles," MS Thesis, research completed, thesis is in process of being written.

Shahi, K. and Wagner, J. B., "Ionic Conductivity and Thermoelectric Power of Pure and Alumina-Dispersed AgI," Abstract No. 134, Electrochemical Society Meeting, Los Angeles, California, October 1979.

Chang, M. R. W., Shahi, K., et al., "Electrical Conductivity of the Solid Electrolyte System  $\text{CuCl}(\text{Al}_2\text{O}_3)$ ," Abstract No. 367, Electrochemical Society Meeting, Boston, Massachusetts, May 6-11, 1979.

Shahi, K. and Wagner, J. B., "Electrical Conductivity of Solid AgI Containing a Dispersion of Submicron Size  $\text{Al}_2\text{O}_3$ ," Abstract No. 369, Electrochemical Society Meeting, Boston, Massachusetts, May 6-11, 1979.

Wagner, J. B., "The Influence of a Dispersed Second Phase on Ionic Conduction in Solid State Electrolytes," AIAA 17th Aerospace Sciences Meeting, New Orleans, Louisiana, January 1979.

Wagner, J. B., "Transport in Compounds Containing a Dispersed Second Phase," presented at Second Joint Japan-U.S. Seminar, "Transport in Non-Stoichiometric Compounds," sponsored by the Japan Society for the Promotion of Science and the U.S. National Science Foundation, Honolulu, Hawaii, October 10-12, 1979.

Shahi, K. and Wagner, J. B., "Fast Ion Transport in Silver Halide Solid Solutions and Multiphase Systems: The Effect of Strain," submitted to Phys. Rev. Letters, January 1980.

Shahi, K. and Wagner, J. B., "Ionic Conductivity and Thermoelectric Power of Pure and  $\text{Al}_2\text{O}_3$ -Dispersed AgI," submitted to J. Electrochem. Soc., December 1979.

Shahi, K. and Wagner, J. B., "The Effect of Process Variables on the Enhanced Conductivity of AgI and CuCl Containing Dispersed  $\text{Al}_2\text{O}_3$  Particles," submitted for possible presentation at 157th Electrochemical Society Meeting, St. Louis, Missouri, May 11-16, 1980.

## Foam Copper

Marvalaud, Inc.

Professor R. B. Pond

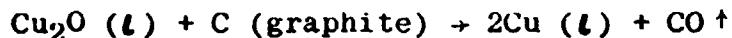
John M. Winter

NAS8-33021 \$40K/year

April 27, 1978 - October 15, 1980

In one "g" gas bubbles move so quickly out of molten metals that it is not feasible to solidify a true "foam." In microgravity such density-driven flow can be insignificant. It may be feasible, therefore, to entrap gas bubbles during solidification in microgravity, resulting in a metal "foam." The objective of this work is to implement a microgravity experiment to determine if such an idea will work.

The idea is to use the reaction



to generate gas bubbles in a melt after it reaches microgravity. A deoxidized copper specimen is prepared with a homogeneous dispersion of fine graphite and a separate source of copper oxide. At 1100°C to 1150°C, only the graphite remains solid, serving as nucleation sites for gas bubbles. The whole experiment is contained at a positive pressure which is appropriate for the desired bubble size.

## Dendritic Solidification at Small Supercoolings

Rensselaer Polytechnic Institute  
Dr. M. E. Glicksman  
NAS8-32425 \$119K (approx.)  
March 1, 1977 - December 15, 1980

The primary objectives of this research task are:  
(1) to obtain reliable kinetic and morphological data on supercoolings which will provide a firmer basis for critically testing current theories and assessing the relative roles and interactions of diffusive and convective transport processes during solidification on Earth, and (2) to study convection-free dendritic growth at low solidification rates. This will provide an opportunity to study micro-morphological details such as side-branch evolution and the crystallographic influences of the tri-axial anisotropy of the solid-liquid surface energy.

Equipment and methods have been established to purify succino-nitrile in fractional kilogram amounts to at least 6-9's purity, and to effect transfer to an apparatus for kinetic and morphological measurements without loss of purity. A combination of vacuum distillation and zone refining is employed based on prior experience of the Principal Investigator. A "universal" stage has been developed to permit full rotation about a vertical axis and limited tilt of  $\sim 10^\circ$  about a horizontal axis of the specimen chamber within the thermostat. This stage permits studies of the influence of the orientation of the gravity vector on the kinetics and morphology of dendritic growth.

### Publications

Glicksman, M. E., "Dendritic Solidification - Myths, Models, Measurements," The Metal Science Club of New York, New York City, New York, April 7, 1977.

Glicksman, M. E., "Capillary Phenomena During Solidification," 5th International Conference on Crystal Growth, Cambridge, Massachusetts, July 19, 1977.

Glicksman, M. E., "Convective Heat Transfer During Dendritic Solidification," AIAA 16th Aerospace Sciences Meeting, Huntsville, Alabama, January 16-18, 1978.

Glicksman, M. E., "Advances in Solidification Fundamentals," Sixteenth George Sachs Seminar on Advances in Foundry Technology, ASM Chapter, Syracuse, New York, April 11, 1978.

Glicksman, M. E., "Melting and Solidification: Microscopic Considerations and Single Phase Systems," ASM Symposium on Melting and Solidification of Metals: Theory and Practice, General Electric R&D Center, Schenectady, New York, May 23, 1978.

Glicksman, M. E., "Gravity-Induced Convection During Dendritic Solidification," COSPAR XXI, Innsbruck, Austria, June 6, 1978.

Glicksman, M. E., "Influence of Spatial Orientation on the Kinetics of Convective-Diffusive Dendritic Growth," Fourth American Conference on Crystal Growth, NBS, Washington, D.C., July 18, 1978.

Glicksman, M. E., "Convective Heat Transfer During Dendritic Growth," 15th Meeting of Society of Engineering Science, Gainesville, Florida, December 4-6, 1978.

Glicksman, M. E., "Convection and Diffusion Effects During Dendritic Solidification," AIAA 17th Aerospace Sciences Meeting, New Orleans, Louisiana, January 15-17, 1979.

Glicksman, M. E., "Development of 'Scaling Laws' and Their Application to Understanding Microstructural Evolution During Solidification," Petrie Lecture, Materials Engineering Department, Technion, Haifa, May 18, 1979.

Glicksman, M. E., "Convective Flow During Dendritic Growth," Fluids Experiment System Workshop, NASA/MSFC, Huntsville, Alabama, July 12, 1979.

Glicksman, M. E., "Scaling Laws in Solidification," Connecticut Chapter of AIME, Wallingford, Connecticut, October 11, 1979.

Glicksman, M. E., "Influence of Metal Convection on the Kinetics of Dendritic Solidification," Olin Metals Research Laboratory, New Haven, Connecticut, October 12, 1979.

Glicksman, M. E., "Convection and Solidification Processes," Department of Mechanical and Aerospace Engineering Colloquium, Case Western Reserve University, Cleveland, Ohio, November 14, 1979.

Glicksman, M. E., "Convective Heat Transfer During Dendritic Growth," Symposium on Fluid Mechanics and Materials Processing, 15th Annual Meeting of the Society of Engineering Science, Gainesville, Florida, December 6, 1979.

Glicksman, M. E. "Capillary Phenomena During Solidifications," J. Cryst. Growth, 42, 1977, pp. 347-356.

Glicksman, M. E. and Huang, S. C., "Transport Phenomena in Dendritic Growth Processes," Proceedings of 87th AIChE Meeting, Boston, Massachusetts, August 1979.

Glicksman, M. E. and Huang, S. C., "Convective Heat Transfer During Dendritic Growth," Third Euro. Sym. on Mat. Sci. in Space, Grenoble, France, April 1979, ESA SP-142.



## Solidification Processes Involving Solutes

Rensselaer Polytechnic Institute  
Dr. M. E. Glicksman  
NAS8-32690      \$82K/year (approx.)  
July 24, 1978 - June 30, 1980

The primary objective of the proposed investigation is to achieve quantitative understanding of the effect of solute additions on the growth kinetics and morphology during dendritic growth under terrestrial gravitational conditions. The data so obtained will help provide fundamental knowledge of the relationship of dendrite morphology to solute content, including especially the influence of melt solute content on the structure and growth rate kinetics. From this study and others, one can eventually determine the range and degree of influence that a low-gravity and nearly convection-free environment will have on the structure of solidified alloys.

Techniques for materials preparation, characterization, and encapsulation will be employed to prepare high-purity succinonitrile as a solvent system. A suitable solute or solutes will be found to "dope" the solvent system to various solute levels. A major emphasis will be placed on obtaining in situ high-resolution photographs of dendritic crystals growing at various concentration levels. This information gives direct structural evidence on the morphological influence of a solute. Emphasis also will be placed on measurements of axial growth rate kinetics for dendritic crystals growing under a variety of experimental conditions.

### Solutal Convection During Directional Solidification

National Bureau of Standards  
Dr. S. R. Coriell  
H-27954B \$80K/year (approx.)  
April 1977 - continuing task

The objective of this task is to calculate and measure effects of convection caused by simultaneous temperature and concentration gradients on directional solidification, including determination of segregation effects in experiments done on Earth and estimation of the effect of microgravity and magnetic fields in avoiding such convection.

Theoretical methods are being developed to predict convective and interfacial instabilities during unidirectional solidification of binary alloys in the vertical direction. Linear hydrodynamic stability theory incorporating solidification boundary conditions has been formulated and has established some criteria for the onset of nonplanar solidification and nonuniform incorporation of solute in the resulting solid, including effects of magnetic fields. This research will delineate the conditions under which gravity-driven convection will occur during constant velocity unidirectional solidification.

#### Publications

Boettinger, W. J., Coriell, S. R., et al., "NBS: Properties of Electronic Materials," NBSIR 78-1483, edited by J. R. Manning and R. L. Parker, 1978, pp. 15-68.

Boettinger, W. J., Coriell, S. R., et al., "NBS: Properties of Electronic Materials," NBSIR 79-1767, edited by R. J. Manning, 1979, pp. 25-81.

Coriell, S. R., Cordes, M. R., et al., "Convective and Interfacial Instabilities During Unidirectional Solidification of a Binary Alloy," J. Cryst. Growth, 49, May 1980, pp. 13-28.

## Direct Observation of Interface Stability

Stanford University

Professor W. A. Tiller

Professor R. S. Fiegelson

Dr. Dennis Elwell

NAS8-33110 Total Cost: \$165K (approx.)

December 1, 1978 - October 31, 1980

This task will use a direct observation method to facilitate the careful test of interface stability criteria in a convective environment. This study will form the basis for future experiments under nonconvective conditions. The objective is a careful test of theory with experiment on a model system with all the significant material parameters being measured for this system. Such a careful test has not been performed heretofore.

The relevant parameters will be measured, including (1) diffusion coefficients of the solutes in the liquid, (2) phase diagram and effective distribution coefficients for the solute, (3) liquidus slopes for the chosen solutes, (4) thermal conductivities and diffusivities for both liquid and solid, (5) solid-liquid interfacial energy and its coefficient of anisotropy, and (6) a study of interface attachment kinetics as a function of orientation.

### Publications

Tiller, W. A., "Interface Field Effects on Solute Redistribution During Crystallization," J. Cryst. Growth, 1980.

## **C. GLASSES, CERAMICS, AND REFRACTORIES**

## Containerless Processing of Optical Glass in Space

Rockwell International

Ralph Happe

NAS8-32023      Total Cost: \$624K (approx.)

April 1979 - April 1983

The overall program task is to increase the knowledge of containerless melting and cooling of oxides or other related inorganic compounds. It is expected that containerless melting and cooling will minimize the possibilities of heterogeneous crystal nucleation on cooling from the melt by eliminating such crystal nucleation sites as container walls and other crystalline material. Such suppression of heterogeneous nucleation should enhance glass-forming possibilities and permit the preparation of glasses in bulk form from materials whose low viscosity prevents such preparation in massive form by terrestrial processes. Containerless processing also promises to permit the preparation of glasses from substrates whose melting points and/or reactivity is too high to permit melting in any known container material.

Terrestrial research has been and will continue to be performed, leading to flight experiments aboard sounding rockets (SPAR) and the Space Shuttle. The terrestrial research relies heavily on a technique of air suspension with CO<sub>2</sub> laser melting developed by the Principal Investigator to identify promising compositions for space research. Among such compositions identified to date are binary gallia-calcia and the higher melting alumina-calcia and lanthana-alumina-calcia compositions.

Following screening of candidate compositions, further terrestrial research will be performed with the most promising compositions to determine suitable processing techniques for preparing samples for space research aboard sounding rockets, early Space Shuttle flights and, in more sophisticated equipment, on later Shuttle/Spacelab flights.

### Publications

Happe, R. A., Implications of Zero Gravity for Producing New Glasses in Space," J. Non-Cryst. Solids, 3, 375 (1970).

Happe, R. A., "Possibilities for Producing New Glasses in Space," Space Processing and Manufacturing, ME 69-1, NASA/MSFC, October 21, 1969, 30 pgs.

Happe, R. A., "Study of the Production of Unique New Glasses," NAR, Downey, California, NAS8-28014, NASA CR-123740, June 1975, 153 pgs.

Happe, R. A., "Manufacturing Unique Glasses in Space," Rockwell International, NAS8-28991, First Interim Report, April 30, 1973-November 1, 1974, 77 pgs.

Happe, R. A., "Manufacturing Unique Glasses in Space," Rockwell International, NAS8-28991, Second Interim Report, March 31, 1976, 38 pgs.

Happe, R. A. and Topol, L. E., "Oxide Glass Processing," Proceedings of Third Space Processing Symposium: Skylab Results, Vol. II, M-74-5, June 1974, pp. 887-924.

Happe, R. A., "The Glass Formation Regions of Oxide Mixtures Based on the Oxides of Aluminum, Cerium, Lanthanum, Magnesium, Niobium, Tantalum, Titanium, Zinc and Zirconium (Hafnium)," Technical Report SD74-SA-0021, Space Division, Rockwell International. (This is a literature search compiled in early 1971 and contains listings of glass formation regions for the above oxide systems from the open literature, with emphasis on the period 1965-1970. There are 121 references cited.)

Happe, R. A. and Topol, L. E., "Experiments Leading to the Production of New Glasses in Space," AIAA 12th Aerospace Sciences Meeting, February 1, 1974, AIAA Paper 74-159.

Topol, L. E., et al., "Formation of New Oxide Glasses by Laser Spin Melting and Free Fall Cooling," J. Non-Cryst. Solids, 12, 377 (1973).

Topol, L. E., et al., "Formation of New Lanthanide Oxide Glasses by Laser Spin Melting and Free Fall Cooling," J. Non-Cryst. Solids, 15, 116 (1974).

Ultrapure Glass Optical Waveguide Development in  
Microgravity by the Sol-Gel Process

Battelle Columbus Laboratories  
Dr. S. P. Mukherjee  
JPL 955361 Total Cost: \$74K (approx.)  
March 1979 - September 1979

The ultimate objective of the present research is to take advantage of the containerless melting of glasses in space for the preparation of ultrapure homogeneous glass for optical waveguides. However, the homogenization of the glass using conventional raw materials is normally achieved on Earth either by the gravity-induced convection currents or by the mechanical stirring of the melt. Because of the absence of gravity-induced convection currents, the homogenization of glass using conventional raw materials will be difficult in the space environment.

Prior to "space readiness", several critical control points must be carefully evaluated on Earth. Hence, the present ground-based program plan is comprised of two major phases:

Phase 1 involves the development of the gel preparation in multicomponent systems and characterization of glasses obtained from gels.

Phase 2 will involve (a) the preparation of ultrapure gels, (b) the melting of ultrapure gels using both a container and containerless melting, and (c) characterization and evaluation of glasses thus obtained.

Publications

Mukherjee, S. P., "Significance and Applications of the Sol-Gel Processes in Glass Industry in Space, presented at the Workshop on Glass and Ceramic Industries in Space Based on Lunar Materials at the Lunar and Planetary Institute, Houston, Texas, to be published in Space Solar Power Review.

Mukherjee, S. P., "Preparation and Characterization of Gels in the  $\text{Na}_2\text{O}-\text{B}_2\text{O}_3-\text{SiO}_2$  System," to be submitted for publication.

## Fining of Glasses in Space

Jet Propulsion Laboratory

Dr. M. C. Weinberg

NAS7-100 Total Cost: \$550K (approx.)

July 1978 - July 1983

The major objective of this study is to investigate the relative importance of dissolution and buoyant effects and to ascertain the role played by refining agents in bubble elimination from glass melts. Also, the nucleation of gas bubbles in melts will be considered.

A research program has been initiated which consists of two major components. Theoretical studies have begun, with the ultimate aim being to mathematically model the refining process. The approach consists of considering increasingly more complicated bubble dissolution (growth) problems. Experimental studies have been performed to determine the velocity of a rising gas bubble in a soda-lime melt. Also, some preliminary experiments have been performed to determine refining rates in soda-lime melts. Additional single bubble dissolution experiments and refining experiments are planned.

### Publications

Weinberg, M. C., Onorato, P. I. K., et al., "The Behavior of Bubbles in Glass Melts: I. Dissolution of a Stationary Bubble Containing a Single Gas," to appear in J. American Ceramic Society, March-April 1980.

Weinberg, M. C., Onorato, P. I. K., et al., "The Behavior of Bubbles in Glass Melts: II. Dissolution of a Stationary Bubble Containing a Diffusing and a Non-Diffusing Gas," to appear in J. American Ceramic Society, July-August 1980.

Subramanian, R. S. and Weinberg, M. C., "The Role of Convective Transport in the Dissolution or Growth of a Gas Bubble," submitted to J. Chemical Physics.

Zak, M. and Weinberg, M. C., "Multibubble Dissolution in Glass Melts," Accepted for publication in J. Noncryst. Solids.

Weinberg, M. C. and Subramanian, R. S., "Dissolution of Multicomponent Bubbles," submitted to J. American Ceramic Society.



Zak, M. and Weinberg, M. C., "Gas Diffusion in Multibubble Systems," submitted to Chem. Engr. Sci.

Weinberg, M. C., "Surface Tension Effects in Gas Bubble Dissolution and Growth," submitted to Chem. Engr. Sci.

Weinberg, M. C. and Zak, M., "Gas Bubbles in Glass," AIAA 17th Aerospace Sciences Meeting, New Orleans, Louisiana, January 1979.

Weinberg, M. C., Onorato, P. I. K., et al., "The Fining of Glasses," American Ceramic Society 81st Annual Meeting, Cincinnati, Ohio, May 1979.

Weinberg, M. C., "The Gas Bubble Problem," Chemical Engineering Seminar, University of Rochester, February 1980.

Weinberg, M. C., "The Gas Bubble Problem," Chemical Engineering Seminar, Clarkson College, February 1980.

Weinberg, M. C. and Subramanian, R. S. "Dissolution of Multicomponent Bubbles in Glass Melts," American Ceramic Society 82nd Annual Meeting, Chicago, Illinois, April 1980.

Zak, M. and Weinberg, M. C., "Multibubble Dissolution in Glass Melts," XII International Congress in Glass, July 1980.

Subramanian, R. S. and Weinberg, M. C., "Asymptotic Expansions in Gas Bubble Dissolution," AIChE 73rd Annual Meeting, Chicago, Illinois, November 1980.

## Physical Phenomena in Containerless Glass Processing

Clarkson College of Technology

Dr. R. Shankar Subramanian

Dr. Robert Cole

NAS8-32944 Total Cost: \$554K (approx.)

December 1977 - December 1982

The objective of this work is to study the behavior of gas bubbles inside drops of model fluids and molten glasses in free fall, focusing on their migration and interaction. Such migration will be induced by thermocapillarity, rotation and/or oscillation of the drop, and other means. The results of the experiments are expected to be of use in the development of techniques for mixing and fining glasses in space and in providing a better understanding of how microballoons are formed.

A broad ground-based investigation into the various physical phenomena of importance in the space experiments is under way. Theoretical models of thermocapillary flow in drops, thermal migration of bubbles in drops, and the migration of bubbles in rotating liquid bodies are being developed. Experiments have been conducted on the migration of a bubble to the axis of a rotating liquid body and the rise of bubbles in molten glass. Experiments on thermocapillary motion in pendant drops and shallow pools of liquid, the influence of oscillation on bubble migration, and volatilization phenomena in glasses are under way.

### Publications

Subramanian, R. S. and Chi, B., "Bubble Dissolution with Chemical Reaction," in press.

Weinberg, M. C. and Subramanian, R. S., "Dissolution of Multicomponent Bubbles," submitted to J. American Ceramic Society.

Subramanian, R. S. and Weinberg, M. C., "The Role of Convective Transport in the Dissolution or Growth of a Gas Bubble," submitted to J. Chemical Physics.

Subramanian, R. S. and Weinberg, M. C., "Asymptotic Expansions in Gas Bubble Dissolution," in preparation for submission to AIChE Journal.

Annamalai, P., Subramanian, R. S., et al., "The Migration of a Bubble in a Rotating Liquid," in preparation for submission to the ASME J. of Applied Mechanics.

Jayaraj, K., Cole, R., et al., "Thermocapillary Motion in a Drop," in preparation for submission to the AICHE Journal.

Shankar, N., Cole, R., et al., "The Quasi-Static Thermocapillary Migration of a Bubble in a Drop," in preparation for submission to the International J. of Multiphase Flow.

Jucha, R. B., Cole, R., et al., "Bubble Rise in Molten Glasses," in preparation for submission to the J. American Ceramic Society.

Subramanian, R. S. and Cole, R., "Space Shuttle Experiments in Glass Processing," AICHE 71st Annual Meeting, Miami Beach, Florida, November 1978.

Subramanian, R. S., and Cole, R., "A Study of Physical Phenomena in Containerless Glass Processing in Space," AIAA 17th Aerospace Sciences Meeting, New Orleans, Louisiana, January 1979.

Subramanian, R. S. and Cole, R., "Space Shuttle Experiments on Containerless Glass Processing," American Ceramic Society 81st Annual Meeting, Cincinnati, Ohio, May 1979.

Annamalai, P., Subramanian, R. S., et al., "Rotation as a Centering Mechanism for Microballoon Fusion Targets," Topical Meeting on Inertial Confinement Fusion, Optical Society of America, San Diego, California, February 1980.

Weinberg, M. C. and Subramanian, R. S., "Dissolution of Multicomponent Bubbles in Glass Melts," American Ceramic Society 82nd Annual Meeting, Chicago, Illinois, April 1980 (accepted).

Subramanian, R. S. and Weinberg, M. C., "Asymptotic Expansions in Gas Bubble Dissolution," AICHE 83rd Annual Meeting, Chicago, Illinois, November 1980, (submitted).

Annamalai, P., Subramanian, R. S., et al., "Bubble Migration in a Rotating Liquid-Filled Sphere," AICHE 73rd Annual Meeting, Chicago, Illinois, November 1980, (submitted).

### Glass Fining Experiments in Zero Gravity

Clarkson College of Technology

Dr. W. R. Wilcox

Dr. R. Shankar Subramanian

Dr. H. D. Smith, Westinghouse

Dr. R. G. Seidensticker, Westinghouse

NAS8-33017 Total Cost: \$266K (approx.)

September 1978 - October 1980

Removal of bubbles (fining) in glasses is a serious problem even on Earth. In the absence of gravity the problem will be even more severe, especially the containerless mode.

A dual approach is being taken for this task: (1) Westinghouse is constructing a SPAR apparatus designed to do experiments on bubbles in molten borax. Bubble behavior will be recorded via a 35 mm camera attached to suitable lenses to provide a closeup view. The apparatus is ready for flight. (2) Clarkson is performing analytical and numerical studies of single-bubble and double-bubble behavior. Significant progress has been made, although no pieces of work have yet been completed.

**Crystal Nucleation in Glass-Forming Alloy and Pure Metal  
Melts Under Containerless and Vibrationless Conditions**

Harvard University  
Professor David Turnbull  
NAS8-32691      Total Cost: \$120K (approx.)  
June 23, 1978 - June 22, 1981

The main objective of this research is to characterize nucleation behavior in glass-forming alloy melts. Such experiments should indicate if formation of alloy glasses in bulk form is possible and, if so, what are the necessary conditions. The most favorable conditions would be those in containerless, vibrationless experiments in high vacua or inert atmospheres.

The crystal nucleation behavior of  $\text{Au}_4\text{Si}$  and  $\text{Pd}_4\text{Si}$  glass-forming melts and of pure Ni by the droplet technique is being investigated. It has been found that the onset undercooling,  $\Delta T_0$ , for copious nucleation in molten  $\text{Au}_4\text{Si}$  droplets varies widely with thermal treatments which alter the nature of the  $\text{SiO}_2$  film on the droplet surface. However,  $\Delta T_0$  as large as 1/3 of the liquidus temperature for some droplets was observed. Glass and crystallization temperatures of  $\text{Au}_4\text{Si}$ -based alloys are sharply increased ( $\sim 1^\circ$  per atom %) when Cu replaces some of the Au.

**Publications**

Turnbull, D., "On Anomalous Prefactors from Analysis of Nucleation Rates," NASA Quarterly Report, September 1979.

Spaepen, F. and Turnbull, D., "Negative Pressures and Melting Point Depression in Oxide-Coated Liquid Metal Droplets," Scripta Met., Vol. 13, 1979, p. 149.

Thompson, C. V. and Spaepen, F., "On the Approximation of the Free Energy Change on Crystallization," Acta Met., Vol. 27, 1979, p. 1855.

A Proposal to Measure the Viscosity of Molten  $\text{Pd}_{78}\text{Si}_{16}\text{Cu}_6$  —  
A Critical Experiment to Support Possible Space Flights

Drexel University  
Dr. Arthur E. Lord  
NAS8-3372      \$29K/year (approx.)  
February 1979 - June 1980

Metallic glasses are materials that are cooled very rapidly to bypass crystallization. Due to the lack of crystallinity, these materials have very unique properties (very "soft" magnetic properties, high hardness, high yield stress, corrosion resistance, etc.) In general, only small sizes are available. To try and make larger samples the time-temperature transformation (TTT) curves must be determined in order to calculate the cooling rate ( $R_c$ ) needed for glass formation. The most important physical property needed for the TTT diagram calculation is the viscosity and its temperature dependence.

An oscillating crucible method for determining the viscosity of molten  $\text{Pd}_{78}\text{Si}_{16}\text{Cu}_6$  alloy has been developed. This material is one of the easiest glass formers and hence can be made in bulk. The results for the auxiliary eutectic alloy  $\text{Pd}_{82}\text{Si}_{18}$  indicate an extremely high viscosity about 50 poise at the melting point. This is about 1000 times the value for other liquid metal systems. The viscosity of  $\text{Pd}_{78}\text{Si}_{16}\text{Cu}_6$  is presently being determined, and with these data the calculations for the best TTT diagram viscosity will be obtained. Interesting results have also been obtained for the viscosity of Fe-Ni-P-B alloys.

Publications

Steinberg, J. and Lord, A. E., "The Viscosity of Molten Fe-Ni-P-B Alloys," J. Noncryst. Solids (submitted for publication).

Steinberg, J. and Lord, A. E., "The Viscosity of Molten  $\text{Pd}_{82}\text{Si}_{18}$  Alloy," J. Noncryst. Solids (submitted for publication).

Lord, A. E., Wouch, G., and Frost, R. T., "Preparation of Amorphous Ferromagnetic Materials through Containerless Solidification," SPAR IV Report, NASA TM-78235, January 1980.

## A Thermochemical Study of Corrosive Reactions in Oxide Materials

National Bureau of Standards  
Dr. H. S. Parker  
H-27954B \$80K/year (approx.)  
April 1977 - April 1980

The objectives of this task are to investigate the nature, extent and limitations on experimental studies imposed by sample container reaction at high temperature and to evaluate containerless techniques for melt and solid state investigations.

The general approach taken was to select a system of current technological importance and investigate: (a) maximum temperature limits for the container materials and characterize the reaction products, (b) determine the equilibrium phase assemblages in selected portions of the system within the limitations imposed by (a), and (c) in collaboration with MSFC provide specimens for levitation experiments at MSFC and correlate the results of levitation experiments with container experiments to evaluate problems associated with low gravity experiments.

The system being investigated was the  $\text{FeO-iron oxide-SiO}_2$  system. This system is of importance to the MHD effort, where these oxides are the major components of seed and coal slag. The first portion investigated consisted of compositions on the  $\text{KFeO}_2\text{-Fe}_2\text{O}_3$  binary. Because of the high temperatures involved ( $T > 1500^\circ\text{C}$ ), platinum was chosen as the container material. As a result of this study, it was discovered that, contrary to the expected result, platinum was a suitable container, provided most of the Fe was kept in the 3+ state. Also, the existence of an alumina structure-type phase was reported.

### Publications

Parker, H. S., Roth, R. S., et al., "NBS: Properties of Electronic Materials," NBSIR 78-1483, edited by J. R. Manning and R. L. Parker, 1978, pp. 84-100.

Parker, H. S., Roth, R. S., et al., "NBS: Properties of Electronic Materials," NBSIR 79-1767, edited by J. R. Manning, 1979, pp. 82-102.

## Containerless Processing Technology

NASA Marshall Space Flight Center

Dr. W. A. Oran

L. H. Berge

B. B. Ross

Dr. J. E. Rush, UAH

In-House Cost: N/A

The objective of this program is to develop the research area of containerless processing in a microgravity environment, including: (1) the organization of working groups of experts and potential users to help develop a ground-based research program and define possible flight hardware, (2) the development and, in part, definition of the limitations of various ground-based containerless processing techniques, and (3) the conduction of precursory experiments in these ground-based assemblies.

Single-axis acoustic levitation devices were utilized as a basis for developing a system to containerlessly process nonconducting materials. However, there are apparent difficulties in operating one of these devices in a ground-based processing system. If one attempts to spot heat a levitated specimen to a high temperature in a relatively cold gaseous medium, instabilities are apparently established in the acoustic standing wave field which result in the sample being ejected from the levitation well. Acoustic streaming influences the temperature of a specimen levitated in an "isothermal" furnace. The streaming pumps cooler air into the levitation region in a manner such that the temperature of the specimen appears influenced more by the transducer temperature than by the wall temperature.

A study of the use of airjet levitation devices to process nonconducting materials has begun. Airjet systems have been used to containerlessly process metals, although the EM generator was used for heating and the resulting field greatly helped to stabilize the metal when molten.

### Publications

Oran, W. A., "Proceedings of the Workshop on an Electromagnetic Positioning System in Space," NASA CP-2069, October 1978.

Oran, W. A., "Preliminary Characterization of a One-Axis Acoustic System," NASA TM-78213, January 1979.



Oran, W. A., "A Parametric Study of an Acoustic Levitation System," Rev. Sci. Instrum. 51, 626 (1980).

Oran, W. A., "Some Limitations in Processing Materials in Acoustic Levitation Devices," IEEE Transactions on Ultrasonics, September 1979, pp. 482-486.

Oran, W. A., "Containerless Melting and Solidification," Proceedings of Electrochemical Society Meeting, St. Louis, May 1980.

Oran, W. A., "Containerless Processing of Metals with an Electromagnetic Levitation System," in preparation.

Science Requirements Definition Study for the Electro-  
magnetic Containerless Processing Module (ECPM)

General Electric Company  
Dr. R. T. Frost  
Dr. W. A. Oran, MSFC  
NAS8-33421      \$130K/year (approx.)  
April 9, 1979 - May 31, 1980

The objective of this program is to define the science requirements for containerless processing experiments and the facilities required to carry out these experiments. Facilities considered should include a ground-based facility, extensions possible for high-temperature thermophysical property measurements using a KC-135-mounted facility and a Shuttle-borne ECPM.

The collection and analysis of science requirements is carried out through personal liaison with investigators active in topics related to containerless processing and through periodic workshops. The individual experiments requirements are then translated into requirements for experiment system components capable of fulfilling most experiment requirements. The work includes a conceptual definition for both an airplane-borne and a Shuttle-borne experiment facility.

Publications

Minutes of the Electromagnetic Containerless Processing Task Team Meetings, Valley Forge Space Center, April 20, 1979, and Marshall Space Flight Center, October 16-17, 1979.

Thermodynamic Properties of Refractory Inorganic Materials  
at High Temperatures

National Bureau of Standards  
J. H. Colwell  
H-27954B      \$50K/year (approx.)  
April 1978 - continuing task

The objective of this research is to develop containerless techniques for determining thermophysical properties of nonmetallic samples at high temperatures.

Two approaches have been considered for carrying out the low-gravity experiments. The first is analogous to a drop-calorimetry experiment in that the sample would be heated to a predetermined temperature whereupon its enthalpy would be determined by engulfing the sample in a calorimeter. This approach would be tedious since a single point would be obtained for each sample, and many points would be needed for the requisite data. The second approach that is presently being pursued involves heating a sample to a high temperature and then allowing it to freely cool while simultaneously measuring its surface temperature and total radiant energy loss as a function of time. Temperature gradients within the samples will complicate the interpretation of the experiments but will also permit thermal conductivities of the samples to be determined under certain conditions. The limiting parameters for these measurements are being determined by computer modelling experiments. Pyroelectric detectors are being tested for potential use in the measurement of the total radiation from the samples.

Publications

Colwell, J. H., "NBS: Properties of Electronic Materials," NBSIR 79-1767, edited by J. R. Manning, 1979, pp. 103-131.

## Fusion Target Technology

Jet Propulsion Laboratory  
Dr. T. G. Wang  
In-Center \$300/year  
October 1979 - continuing task

The objectives of this task are to (1) study the physical processes that are associated with the fabrication of inertial confinement fusion (ICF) targets in a weightless environment, (2) determine jointly with DOE centers the need for extended zero gravity in the future production of ICF targets, and (3) provide technological information to DOE centers that is pertinent to their current target fabrication research.

To produce the high-quality fusion target shells that are required, four fundamental physical processes must be understood: spheroidization of the shell, uniformity of shell thickness and coating, adiabatic expansion, and contraction of the molten pellet as it passes through temperature gradient environments.

The approach to be taken is to (1) study the fluid dynamic processes that pertain to pellet fabrication processes such as bubble centering, coating uniformity, and various instabilities; (2) study the effects of various temperature levels and temperature gradients on pellet fabrication; (3) construct Earth-based high temperature and high temperature gradient drop towers; (4) initiate development of a process for the studies, numerical analyses, and computer calculations on various pellet fabrication processes.

### Publications

Wang, T. S., "Gravitational Effects on Target Fabrication," Technical Digest of the Conference on Inertial Confinement Fusion, San Diego, California, February 1980.

Elleman, D. D., Croonquist, A. P., et al., "Low Gravity Acoustic Positioning Chamber for Liquid Shell Studies," Technical Digest of the Conference on Inertial Confinement Fusion, San Diego, California, February 1980.

Lee, M. C., Helizon, R. S., et al., "Real-Time Image Acquisition and On-line Image Processing of a Hollow Sphere Confined in a Drop Furnace," Technical Digest of the Conference on Inertial Confinement Fusion, San Diego, California, February 1980.

Kendall, J. M., "Fluid Dynamics Relating to the Production of Large Fusion Targets," Technical Digest of the Conference on Inertial Confinement Fusion, San Diego, California, February 1980.

## Glass Shell Manufacturing in Space

KMS Fusion, Inc.

Dr. Robert L. Nolen

NAS8-33103 Total Cost: \$266K (approx.)

December 6, 1978 - December 5, 1981

The principal objective of this research is to develop a detailed understanding of the chemical and physical processes involved in the formation of uniform, high-quality spherical glass shells. Where possible, various stages of the shell-blowing process will be formulated into mathematical models.

The temperature regimes where major transformations occur in the shell starting materials (metal-organic gels) have been identified. This was accomplished using a combination of thermal analytical techniques. The gases generated by pyrolysis of the gel were quantitatively characterized by gas chromatography and pressure tests.

A mathematical model of the heat transfer mechanisms occurring in the gel-to-shell transformation will be refined from one previously formulated under another contract. Computerization of the mathematical model will be used to bring it into line with the results from a series of controlled drop tower furnace experiments to be done with fully characterized and standardized gel powder pellets. Experiments conducted in the course of modeling the heat transfer process should also provide information on gas transport mechanisms.

### Publications

Nolen, R. L., "Glass Shell Manufacturing in Space," First-Fourth Quarterly Reports, NAS8-33103, March 1979-January 1980.

## The Upgrading of Glass Microballoons

Bjorksten Research Laboratories  
Dr. Stanley A. Dunn  
Robert T. Nagler  
Elmer G. Paquette  
Stephen Gunter  
Dr. E. J. Crosby  
NAS8-33513      \$43.8K/year (approx.)  
August 28, 1978 (repropose each 12 months)

The objective of this program is to study extensively the processes and mechanisms involved in producing glass microballoons of acceptable quality for laser fusion by gas jet levitation and manipulation in the molten condition.

The gas supporting levitation microfurnace (LMF) concept represents a unique means of handling and shaping small bodies where contact with foreign condensed phase objects or supports could be deleterious. Levitation is achieved by aerodynamic lift imparted to the body from a gaseous flow field generated with the aid of a collimated hole structure (CHS). A long-standing objective of the development of the LMF has been the perfecting of glass microballoon (GMB) geometry by centrifugal centering and coordinated temperature and pressure manipulation. Centering by centrifugal means implies the ability to control the orientation of the axis of rotation as well as the speed.

Existing LMF designs provide excellent horizontal and vertical translational stability. A third requirement, that of rotational control has been partially solved. Rate of rotation can be controlled down to very slow speeds. The ability to stop rotation entirely and to index the GMB to any desired orientation are objectives now being sought through division of the field of jets into three or four regions, each with separately controllable gas flows. The capability of being able to stop rotation as well as to control position will be of use in studies of heat transfer involving GMB's.

### Publications

Dunn, S. A. and Gunter, S., "The Upgrading of Glass Microballoons - Targets for Laser Fusion." NASA CR-161280, 1979.

## Advanced Containerless Processing Technology

Jet Propulsion Laboratory

Dr. T. G. Wang

In-Center \$350K/year

October 1978 - continuing task

The primary long-range objectives of this task are to: (1) study and advance the science of contactless positioning and manipulation of a high-temperature acoustic chamber, (2) provide design information on a flight version of this chamber for materials science studies in a contactless and zero gravitation environment, and (3) provide potential MPS investigators with a set of ground-based facilities with which to perform precursor experiments.

The approaches to be taken in FY 81 are experimental and theoretical studies of (1) acoustic positioning and manipulation capabilities of a rectangular chamber as a function of temperature and pressure, (2) various acoustical geometries which may have special application in materials science studies, (3) loss mechanisms associated with high-intensity and high-temperature acoustic waves, (4) an aeroacoustic positioning system which will allow us to levitate heavy samples in the laboratory, (5) a liquid-liquid positioning system which will allow us to study the dynamic of liquid melts, and (6) positioning and manipulation capabilities of a KC-135 acoustic module.

In addition, a new effort will be initiated this year to provide MPS investigators with a set of facilities to perform precursor experiments prior to spaceflight. These facilities will allow investigators to examine and compare the properties of their sample processes in the following four ways: one-g contained, one-g containerless, zero-g contained, and short-duration zero-g containerless.

### Publications

Lagomarsini, G. and Wang, T. G., "Acoustic Containerless Processing Module for Materials Research," AIAA, 79-0369, 1979.

Jacobi, N., Tagg, R. P., et al., "Free Oscillations of a Large Drop in Space." AIAA, 79-0225, 1979.



Lee, M. C., "Temperature Dependence of Molecular Vibrational Relaxation Frequency for  $O_2-H_2$ ,  $O_2-He$  and  $O_2-CO_2$  Mixture Between 300-675°K," Proceedings of the Ultrasonics Symposium, IEEE, New Orleans, September 1979.

Barmatz, M. B., "Nonlinear Effects in a High Intensity Acoustic Resonant Chamber," Proceedings of the 10th International Congress on Acoustics, Sydney, Australia, July 1980.

Trinh, E., Wang, T. G., and Lee, M. C., "A Technique for the Study of Drop Dynamics in a Liquid-Liquid System," JASA 67, S1 (1980).

Wang, T. G., "Acoustic Levitation and Manipulation for Space Applications," Proceedings of the Ultrasonics Symposium, IEEE, New Orleans, September 1979.

Wang, T. G., Elleman, D. D., et al., "Containerless Processing Technology Experiment Report, SPAR IV Experiment 76-20, SPAR IV Final Report, NASA TM-78235, January 1980.

Electrostatic Control and Manipulation of Materials for  
Containerless Processing

Jet Propulsion Laboratory

Dr. M. M. Saffren

Dr. D. D. Elleman

Dr. W. K. Rhim

In-Center \$250K (to date)

October 1978 - continuing task

The objective of this task is to develop electric field positioning/manipulation techniques and technology for the containerless processing of materials in bulk and dispersed forms. This method obviates limitations of other methods. Two principal demonstrations to prove technique and technology will be completed no later than CY 83. An important part of the task is to ensure that facilities to be developed satisfy requirements of identified user applications. Another part of the task is to pursue study of the electrohydrodynamics of liquid drops (that must be done as part of the development) as a fundamental investigation.

Task studies include: methods and limitations of electric field confinement, electrode configuration, electrohydrodynamics of both charged and neutral drop systems, and dynamics and stability of charged particle arrays. These investigations are performed in the laboratory, in the KC-135 aircraft, and through theoretical and numerical study. Use is made of available SPAR flight experiment data, and Shuttle flight experiments are planned.

Publications

Saffren, M. M., Elleman, D. D. and Rhim, W. K., "Dynamics of Liquid Shells," Conference on Inertial Confinement Fusion, San Diego, California, February 26-28, 1980.

Saffren, M. M., Elleman, D. D., and Rhim, W. K., "Dynamics of Compound Liquid Drops," JPL Report 715-27: "Theoretical Development--Concentric Drops--Mode Frequencies and Interfacial Displacements," Vol. I; "Theoretical Development--Energy and Angular Momentum," Vol. II; "Numerical Results," Vol. III; "Non-Concentric Modes and Centering Forces--Theoretical Development," Vol. IV.

## **D. FLUIDS, TRANSPORTS, AND CHEMICAL PROCESSES**

## Transient Thermal Convection in Low-g

NASA Headquarters  
Dr. R. F. Dressler  
In-House  
January 1980 - Continuing

The purpose of this research program is to obtain analytical solutions for transient convection flows for arbitrary low-g excitations with imposed thermal gradient in cylinders and cubes, for both 2-D and 3-D flows.

### Publications

Dressler, R. F., "Transient Thermal Convection During Orbital Spaceflight," submitted for publication in Journal of Crystal Growth.

Dressler, R. F., "Closed-Form Convective Solutions for Periodic G-Jitter," in preparation.

Dressler, R. F., "Approximate 3-D Convective Solutions in Low-G," in preparation.

### Fluid Dynamics Numerical Analysis

Lockheed Missiles & Space Company

Dr. L. Spradley

J. Robertson

NASW-3281      Total Cost: \$98K (approx.)

August 1979 - August 1981

The purpose of this research program is to compute transient thermal convection for cases of importance to Materials Processing in Space. This includes problems too difficult for analytical solutions and also includes verification of ranges of validity of theory developed by Dr. R. F. Dressler.

Lockheed's previously derived LOCAP and GIM codes have been modified and adapted for these tasks. Two-dimensional problems are done on MSFC computers; three-dimensional problems on LaRC's STAR. Work is completed on two-dimensional transients for step-functions for circles and squares. Ranges of validity of Rayleigh number for the Dressler theory have been determined. Two-dimensional transient convection in a sphere idealizing the Lal experiment has been completed. Some stability analyses and the three-dimensional flows have not yet begun.

Production of Large-Particle-Size Monodisperse Latexes  
in Microgravity

Lehigh University

Dr. J. W. Vanderhoff

Dr. F. J. Micale

Dr. M. S. El-Aasser

NAS8-32951      Total Cost: \$418K (approx.)

February 22, 1978 - February 22, 1983

The effort will explore the possibility of performing seeded polymerization in low-g to avoid the problems of creaming and sedimentation as the particles grow and change density. If successful, this could provide a method for routinely producing monodisperse spheres in a size range that is difficult to obtain by other techniques.

Ground-based experiments are continuing to obtain kinetic data and to evaluate alternative methods for growing such spheres. Flight hardware has been defined, and negotiations are under way to produce reactors to fly on early Shuttle flights.

Work has continued with the high swelling ratio of monomer to polymer technique developed in the laboratory. Emphasis has recently been put on the evaluation of newly obtained mercaptans which are used in the pretreatment of latex seed. A correlation is being sought between the molecular weight distribution, as a function of the type and concentration of mercaptan, and the swelling ratio. Work has also been carried out, in a continuing effort, toward an evaluation of the degree of agitation obtained in the laboratory prototype monodisperse latex reactor, LUMLR, as a function of rpm.

Publications

Vanderhoff, J. W., et al., "Heterogeneous Chemical Reactions: Preparations of Monodisperse Latexes," Final Report, NAS8-32399, July 1977.

## Solutal Convection During Directional Solidification

National Bureau of Standards  
Dr. S. R. Coriell  
H-27954B \$80K/year (approx.)  
April 1977 - continuing task

The objective of this task is to calculate and measure effects of convection caused by simultaneous temperature and concentration gradients on directional solidification, including determination of segregation effects in experiments done on Earth and estimation of the effect of microgravity and magnetic fields in avoiding such convection.

Theoretical methods are being developed to predict convective and interfacial instabilities during unidirectional solidification of binary alloys in the vertical direction. Linear hydrodynamic stability theory incorporating solidification boundary conditions has been formulated and has established some criteria for the onset of nonplanar solidification and nonuniform incorporation of solute in the resulting solid, including effects of magnetic fields. This research will delineate the conditions under which gravity-driven convection will occur during constant velocity unidirectional solidification.

### Publications

Boettinger, W. J., Coriell, S. R., et al., "NBS: Properties of Electronic Materials," NBSIR 78-1483, edited by J. R. Manning and R. L. Parker, 1978, pp. 15-68.

Boettinger, W. J., Coriell, S. R., et al., "NBS: Properties of Electronic Materials," NBSIR 79-1767, edited by R. J. Manning, 1979, pp. 25-81.

Coriell, S. R., Cordes, M. R., et al., "Convective and Interfacial Instabilities During Unidirectional Solidification of a Binary Alloy," J. Cryst. Growth, 49, May 1980, pp. 13-28.

## Fluid Dynamics of Crystallization from Vapors

University of Utah - Salt Lake City

Dr. F. Rosenberger

NSG-1534      Total Cost: \$160K (approx.)

June 1, 1978 - May 31, 1980

This program is aimed at obtaining fundamental insight into the complex physiochemical fluid dynamics of closed ampoule vapor crystal growth processes to the extent that a desired set of crystal growth conditions can be designed in advance. A more directly applied part of the program is concerned with the synthesis of ultrapure mercuric iodide and the vapor composition (stoichiometry) required for the growth of mercuric iodide high resolution radiation detector crystals.

A comparison of the analytical and numerical modeling possibilities and their relative merits with respect to accuracy and computation costs has been performed on a horizontal cavity with net diffusive-convective mass transport. The singular boundary conditions that arise in this situation were shown to be of little consequence for the accuracy of the concentration and total flux parameters of interest to crystal growth. As experimental prerequisite laser Doppler studies, an apparatus and seeding technique have been developed which allow for the determination of three-dimensional velocity fields in the range  $10^{-2}$  cm/s to 30 cm/s inside a closed cylinder in a closely controlled temperature gradient. Mass spectroscopic capability is being built up to unambiguously determine the actual composition of some of the "bench-mark" systems and to supplement efforts on the impurity analysis of technologically important solid state materials. Deviation from the "ideal" stoichiometry has been determined iodometrically with a resolution of  $10^{-3}$  mole fractions. These efforts supply the basis for planned vapor phase synthesis of  $\text{HgI}_2$  from the ultrapure elements and for the absolute calibration of ongoing thermogravimetric studies of the vapor-solid phase diagram of the  $\text{HgI}_{2\pm x}$  existence range.

### Publications

Rosenberger, F., "Fluid Dynamics in Crystal Growth from Vapors," J. Physchem. Hydrodynamics, 1, in press.



Markham, B. L. and Rosenberger, F., "Velocity and Concentration Distribution in a Stefan Diffusion Tube," submitted to Int. J. of Heat and Mass Transfer.

Markham, B. L., Greenwall, D. W., et al., "Numerical Modeling of Physical Vapor Transport in Vertical Ampoules," submitted to J. Crystal Growth.

Jhaveri, B. S. and Rosenberger, F., "On Singular boundary Conditions in Diffusive-Convective Mass Transfer Across a Cavity," in preparation.

## Fluid Dynamics and Thermodynamics of Vapor Phase Crystal Growth

Rensselaer Polytechnic Institute

Dr. Heribert Wiedemeier

NAS8-33562 Total Cost: \$254K (approx.)

January 1, 1979 - February 2, 1981

The primary objective of this program is to provide basic mass transport and crystal growth data which, combined with a thorough knowledge of the thermodynamics, will improve the fluid dynamic characterization of vapor transport systems.

The program is concerned with the investigation of the effect of systematic variations (1) of the relative importance of buoyancy-driven convection and (2) of diffusion and viscosity conditions on mass transport and crystal growth. These investigations will be performed in evacuated, closed ampoules for selected temperature gradients and partial pressures of the transport species. The specific experimental tasks include mass transport rate studies as a function of ampoule orientation, geometry, and the effects of inert gas additions. These will be correlated with crystal growth rate and morphology studies. In addition, the analysis of the vapor phase is an essential aspect of this project. The experimental efforts are supported by theoretical studies, including the thermodynamic and fluid dynamic characterization of the gas phase and the estimation of mass transport rates for different diffusive and diffusive-convective conditions. The materials investigated under this program include selected group IV elements and IV-VI compounds and halogens as transport agents. This choice is based on increasing complexity, known thermochemical and structural properties of these materials, and the existence of microgravity results for some of these systems investigated in previous Skylab and ASTP experiments.

### Publications

Wiedemeier, H., "The Effect of Inert Gas on the Chemical Transport Properties of the GeSe-GeI<sub>4</sub> System," submitted for publication.

Wiedemeier, H., "The Effect of Orientation of the Density Gradient with Respect to the Gravity Vector on the Mass Transport Properties of the GeSe-GeI<sub>4</sub> System," submitted for publication.

Wiedemeier, H., "The Influence of Ampoule Geometry on the Mass Transport Properties of the GeSe-GeI<sub>4</sub> System," submitted for publication.

Wiedemeier, H., "The Effect of Homogeneous Chemical Reactions on the Mass Transport of Chemical Vapor Transport Systems," submitted for publication.

## Physical Phenomena in Containerless Glass Processing

Clarkson College of Technology

Dr. R. Shankar Subramanian

Dr. Robert Cole

NAS8-32944 Total Cost: \$554K (approx.)

December 1977 - December 1982

The objective of this work is to study the behavior of gas bubbles inside drops of model fluids and molten glasses in free fall, focusing on their migration and interaction. Such migration will be induced by thermocapillarity, rotation and/or oscillation of the drop, and other means. The results of the experiments are expected to be of use in the development of techniques for mixing and fining glasses in space and in providing a better understanding of how microballoons are formed.

A broad ground-based investigation into the various physical phenomena of importance in the space experiments is under way. Theoretical models of thermocapillary flow in drops, thermal migration of bubbles in drops, and the migration of bubbles in rotating liquid bodies are being developed. Experiments have been conducted on the migration of a bubble to the axis of a rotating liquid body and the rise of bubbles in molten glass. Experiments on thermocapillary motion in pendant drops and shallow pools of liquid, the influence of oscillation on bubble migration, and volatilization phenomena in glasses are under way.

### Publications

Subramanian, R. S. and Chi, B., "Bubble Dissolution with Chemical Reaction," in press.

Weinberg, M. C. and Subramanian, R. S., "Dissolution of Multicomponent Bubbles," submitted to J. American Ceramic Society.

Subramanian, R. S. and Weinberg, M. C., "The Role of Convective Transport in the Dissolution or Growth of a Gas Bubble," submitted to J. Chemical Physics.

Subramanian, R. S. and Weinberg, M. C., "Asymptotic Expansions in Gas Bubble Dissolution," in preparation for submission to AIChE Journal.

Annamalai, P., Subramanian, R. S., et al., "The Migration of a Bubble in a Rotating Liquid," in preparation for submission to the ASME J. of Applied Mechanics.

Jayaraj, K., Cole, R., et al., "Thermocapillary Motion in a Drop," in preparation for submission to the AICHE Journal.

Shankar, N., Cole, R., et al., "The Quasi-Static Thermocapillary Migration of a Bubble in a Drop," in preparation for submission to the International J. of Multiphase Flow.

Jucha, R. B., Cole, R., et al., "Bubble Rise in Molten Glasses," in preparation for submission to the J. American Ceramic Society.

Subramanian, R. S. and Cole, R., "Space Shuttle Experiments in Glass Processing," AICHE 71st Annual Meeting, Miami Beach, Florida, November 1978.

Subramanian, R. S., and Cole, R., "A Study of Physical Phenomena in Containerless Glass Processing in Space," AIAA 17th Aerospace Sciences Meeting, New Orleans, Louisiana, January 1979.

Subramanian, R. S. and Cole, R., "Space Shuttle Experiments on Containerless Glass Processing," American Ceramic Society 81st Annual Meeting, Cincinnati, Ohio, May 1979.

Annamalai, P., Subramanian, R. S., et al., "Rotation as a Centering Mechanism for Microballoon Fusion Targets," Topical Meeting on Inertial Confinement Fusion, Optical Society of America, San Diego, California, February 1980.

Weinberg, M. C. and Subramanian, R. S., "Dissolution of Multicomponent Bubbles in Glass Melts," American Ceramic Society 82nd Annual Meeting, Chicago, Illinois, April 1980 (accepted).

Subramanian, R. S. and Weinberg, M. C., "Asymptotic Expansions in Gas Bubble Dissolution," AICHE 83rd Annual Meeting, Chicago, Illinois, November 1980, (submitted).

Annamalai, P., Subramanian, R. S., et al., "Bubble Migration in a Rotating Liquid-Filled Sphere," AICHE 73rd Annual Meeting, Chicago, Illinois, November 1980, (submitted).

## Fining of Glasses in Space

Jet Propulsion Laboratory

Dr. M. C. Weinberg

NAS7-100      Total Cost: \$550K (approx.)

July 1978 - July 1983

The major objective of this study is to investigate the relative importance of dissolution and buoyant effects and to ascertain the role played by refining agents in bubble elimination from glass melts. Also, the nucleation of gas bubbles in melts will be considered.

A research program has been initiated which consists of two major components. Theoretical studies have begun, with the ultimate aim being to mathematically model the refining process. The approach consists of considering increasingly more complicated bubble dissolution (growth) problems. Experimental studies have been performed to determine the velocity of a rising gas bubble in a soda-lime melt. Also, some preliminary experiments have been performed to determine refining rates in soda-lime melts. Additional single bubble dissolution experiments and refining experiments are planned.

### Publications

Weinberg, M. C., Onorato, P. I. K., et al., "The Behavior of Bubbles in Glass Melts: I. Dissolution of a Stationary Bubble Containing a Single Gas," to appear in J. American Ceramic Society, March-April 1980.

Weinberg, M. C., Onorato, P. I. K., et al., "The Behavior of Bubbles in Glass Melts: II. Dissolution of a Stationary Bubble Containing a Diffusing and a Non-Diffusing Gas," to appear in J. American Ceramic Society, July-August 1980.

Subramanian, R. S. and Weinberg, M. C., "The Role of Convective Transport in the Dissolution or Growth of a Gas Bubble," submitted to J. Chemical Physics.

Zak, M. and Weinberg, M. C., "Multibubble Dissolution in Glass Melts," Accepted for publication in J. Noncryst. Solids.

Weinberg, M. C. and Subramanian, R. S., "Dissolution of Multicomponent Bubbles," submitted to J. American Ceramic Society.

Zak, M. and Weinberg, M. C., "Gas Diffusion in Multibubble Systems," submitted to Chem. Engr. Sci.

Weinberg, M. C., "Surface Tension Effects in Gas Bubble Dissolution and Growth," submitted to Chem. Engr. Sci.

Weinberg, M. C. and Zak, M., "Gas Bubbles in Glass," AIAA 17th Aerospace Sciences Meeting, New Orleans, Louisiana, January 1979.

Weinberg, M. C., Onorato, P. I. K., et al., "The Fining of Glasses," American Ceramic Society 81st Annual Meeting, Cincinnati, Ohio, May 1979.

Weinberg, M. C., "The Gas Bubble Problem," Chemical Engineering Seminar, University of Rochester, February 1980.

Weinberg, M. C., "The Gas Bubble Problem," Chemical Engineering Seminar, Clarkson College, February 1980.

Weinberg, M. C. and Subramanian, R. S., "Dissolution of Multicomponent Bubbles in Glass Melts," American Ceramic Society 82nd Annual Meeting, Chicago, Illinois, April 1980.

Zak, M. and Weinberg, M. C., "Multibubble Dissolution in Glass Melts," XII International Congress in Glass, July 1980.

Subramanian, R. S. and Weinberg, M. C., "Asymptotic Expansions in Gas Bubble Dissolution," AIChE 73rd Annual Meeting, Chicago, Illinois, November 1980.

Glass Fining Experiments in Zero Gravity

Clarkson College of Technology

Dr. W. R. Wilcox

Dr. R. Shankar Subramanian

Dr. H. D. Smith, Westinghouse

Dr. R. G. Seidensticker, Westinghouse

NAS8-33017 Total Cost: \$266K (approx.)

September 1978 - October 1980

Removal of bubbles (fining) in glasses is a serious problem even on Earth. In the absence of gravity the problem will be even more severe, especially the containerless mode.

A dual approach is being taken for this task: (1) Westinghouse is constructing a SPAR apparatus designed to do experiments on bubbles in molten borax. Bubble behavior will be recorded via a 35 mm camera attached to suitable lenses to provide a closeup view. The apparatus is ready for flight. (2) Clarkson is performing analytical and numerical studies of single-bubble and double-bubble behavior. Significant progress has been made, although no pieces of work have yet been completed.



## Contact and Coalescence of Viscous and Viscoelastic Bodies

Massachusetts Institute of Technology  
Professor D. R. Uhlmann  
NAS8-31552 Total Cost: \$128K (approx.)  
November 1, 1975 - July 1, 1980

The program was concerned broadly with developing improved understanding of the processes which take place when viscous and viscoelastic bodies come in contact and coalesce.

In the experimental phase of the investigation, the viscous coalescence of liquid droplets suspended in a neutral buoyant fluid was studied. High-speed movies of the coalescence process were taken and analyzed in detail to establish the rate of growth of the radius of the neck between the particles as a function of time. Space experiments were designed to study the coalescence of both viscous and viscoelastic spheres. Difficulties were encountered, however, with the detachment of droplets from the tips of needles. The detachment problem was analyzed, and approaches to circumventing the problem were initiated.

### Publications

Uhlmann, D. E., Contact and Coalescence of Viscous Bodies, Experiment 74-53, SPAR III Final Report, NASA TM-78137, January 1978.

## Uniform Dispersions by Crystallization Processing

Massachusetts Institute of Technology  
Professor D. R. Uhlmann  
NAS8-31350 Total Cost: \$398K (approx.)  
July 1, 1975 - June 1, 1980

The program was concerned broadly with developing improved understanding of the interaction between second-phase particles and an advancing solidification front and with developing criteria for the solidification processing of two-phase composite materials in space.

In the experimental portion of the work, the critical velocity (i.e., the maximum growth rate for which a particle is pushed by a growing crystallization front) was measured for a variety of particles, ranging in size between 1 and 200 microns in both high and low entropy of fusion ( $\Delta S_f$ ) organic materials. The critical velocity was inversely proportional to melt viscosity, was lower in small  $\Delta S_f$  materials than in high  $\Delta S_f$  materials having the same viscosity, and decreased with increasing particle size. Space experiments were conducted to eliminate the possible frictional effects of the particles against the container walls. These experiments indicated that particle pushing by a crystal-liquid interface does occur under microgravity conditions and that particle pushing in microgravity occurs with characteristics similar to those found in 1-g.

### Publications

Uhlmann, D. R., Aubourg, P. A., et al., "Multiphase Dispersions by Crystallization Processing," COSPAR Space Research, Vol. XIX, Pergamon Press, 1979, pp. 525-529.

Uhlmann, D. R., "Behavior of Second-Phase Particles at Solidification Front," SPAR V Final Report, NASA TM-78275, August 1980.

## Marangoni Effect in Crystal Processing

Arthur D. Little  
Dr. Arthur D. Fowle  
Dr. M. C. Davidson, MSFC  
Dr. A. F. Witt, MIT  
NAS8-32946, Total Cost: \$552K (approx.)  
March 1978 - December 1980

The Spacelab experiment consists of measuring the freezing interface morphology and the velocity and temperature fields on the surface of a molten zone in a cylindrical sample of gallium-doped germanium in a crystal growing configuration. The nominal sample diameter is 0.7 cm, and the pulling rate is variable. Postflight characterization of the space-grown crystals will be made, and the results of the measured experimental variables will be compared with theory.

The experiment will be carried out under conditions in which molecular diffusion-controlled processes can dominate the behavior of the melt because gravity-induced convection is made negligible in the microgravity environment of Spacelab. This processing condition is desirable because the the scientific understanding and control of it would produce an immediate promise for producing better semiconductor, crystal products. However, flows driven by surface tension gradient (Marangoni effect) are predicted to upset the diffusion-controlled process ideal. A major purpose of the experiment, and the theory in support of it, is to examine the nature and importance of the Marangoni effect.

### Publications

Fowle, A. A., "Float Zone Experiments System (FZES) for Spacelab," AIAA 17th Aerospace Sciences Meeting, January 1979.

Fowle, A. A., et al., "Experiments on the Stability of Conical and Cylindrical Liquid Columns at Low Bond Numbers," Third European Symposium on Material Sciences in Space, May 1979.

Mass Transfer in Electrolytic Systems Under Low Gravity Conditions

University of Alabama in Huntsville

Dr. C. Riley

Dr. H. D. Coble

Dr. R. B. Owens, MSFC

Gordon Fisher, INCO

NAS8-33542 \$8K

September 1979 - May 1980

Electrodeposition involves mass transfer from one phase to another. In particular one is concerned with the deposition of materials into the solid phase out of the liquid (solution or suspension) phase. Diffusive and convective flow coupled with deposition result in density gradients that are gravity dependent. We desire to characterize this gravity dependence associated with electrodeposition. When a better understanding of the gravity influence is acquired, it should lead to improved control of variables during electroformation of materials. The overall objective will be the electroformation of materials with improved or more desirable properties.

Miniature electrodeposition cells have been constructed. Simple metal-in/metal-out reactions using tin, cobalt or silver are being utilized as the mass transfer agents. The density flow patterns between electrodes with both a vertical and horizontal configuration will be bench characterized using a laser Schlieren system. The metal-in/metal-out system will obviate gas evolution interference on the observation system. These results will be compared to those determined for the same cells under reduced gravity conditions produced during parabolic, free-fall flights of a KC-135 aircraft.

## Surface Tension-Driven Convection Phenomena

Case Western Reserve University  
Dr. Simon Ostrach  
Dr. J. Adin Mann  
NAS8-33015 \$226K/year (approx.)  
May 8, 1979 - May 8, 1980

Very little detailed information exists on the behavior of free-surface liquid configurations under non-isothermal and/or nonisoconcentration conditions. Because such situations are inherent to the containerless processing of materials, the present research program is directed to developing theoretical and experimental techniques to describe and define the nature of surface-tension gradient induced flows and the associated transport phenomena. Surface and bulk flows generated by temperature and concentration (excess) gradients and their coupling will be determined. In addition, extensive characterization of the surface properties will be performed as an essential input to the detailed studies mentioned previously. The development of new instrumental techniques for determining surface flows is an important aspect of this project.

Proper analysis of free-surface problems requires the determination of the surface shape and temperature distribution from the solution. Because of the complexities associated with these, problems of progressively increasing generality will be studied. In this way the efficacy of various approximate methods, such as variational methods and the method of weighted residuals, will be investigated to help reduce the time and cost required to obtain physically meaningful solutions. By a technique developed as part of the present research program surface-tension gradient flows can be studied in deep liquid layers. Flow structures over a range of parameters for various heating modes will be determined.

Experimental study will be made of the interaction of the two types of flows in a small liquid column supported between two rotating ends. Both co- and counter-rotation will be investigated. Laser light-scattering techniques that detect the ripplon Doppler shift are being used and developed to follow the flow fields at surfaces.

### **Publications**

**Pimputkar, S. M. and Ostrach, S., "Transient Thermocapillary Flow in Thin Liquid Layers," accepted for publication in Physics of Fluids, 1980.**

**Basu, S., "Thermally Induced Interfacial Fluctuations," M.S. Thesis, Case Western Reserve University, summer 1979. Three papers have been prepared from this work for submission during the next few months.**

## Analysis of the Float Zone Process

Massachusetts Institute of Technology  
Professor R. A. Brown  
NSG-7645/Supplement 1

This research effort involves a computational simulation of the fluid and heat transfer that takes place in a float zone crystal growth process. Analytical models are being developed for the melt-solid interface to examine the effects of both natural convection and Marangoni convection. Such models are essential for determining the conditions that must be controlled in order to obtain flat interfaces in either ground-based or spaceflight float zone crystal growth.

Thermocapillary Flows and Their Stability: Effects of  
Surface Layers and Contamination

Northwestern University

Dr. S. H. Davis

Dr. G. M. Homsy, Stanford University

NAS8-33881 Total Cost: \$400K (approx.)

June 1980 -- June 1983

The proposed research concerns the theoretical analysis of the fluid mechanics and heat transfer of motions driven by surface-tension gradients. The object is an understanding of the convection accompanying the process of growing high-quality crystals in a  $\mu$ -g environment. The geometries considered include thin films, deep films and float-zone configurations. The particular aspects addressed are (1) the effects on steady Marangoni flow of contamination and the placement of third-phase films on the melt-gas interface, (2) the prediction of possible instabilities of Marangoni flows of pure melts and, (3) the effects on such instability criteria of contamination and surface films.

Calculation of flow and heat transfer characteristics is possible in the following limits: (1)  $A \rightarrow 0$ , (2)  $M \rightarrow 0$ , (3)  $C \rightarrow 0$ . These asymptotic limits, together with perturbation theory, may be used to analytically describe features of the structure of solutions to this class of problems. These analyses will be used to probe the effects of large classes of insoluble surface films on the steady flows generated by surface-tension gradients. It should be possible to describe what film characteristics are required to either suppress convection entirely, retard it to a given degree or even possibly augment it. The types of effects attributable to contamination would be determined. The magnitude of such effects could be calculated given experimental values for certain appropriate physical constants describing the material response of the contaminants.



## Investigation of the Free Flow Electrophoresis Process

McDonnell-Douglas Astronautics Company

R. A. Weiss

D. W. Richman

J. W. Lanham

C. D. Walker

NAS8-33200 Total Cost: \$113K (approx.)

December 12, 1978 - June 4, 1980

Another means of obtaining electrophoretic separation of cells or macromolecules is the use of continuous flow electrophoresis (CFE). By its very nature, free-flow electrophoresis is associated with significant problems, many of which are gravity-determined.

An improved continuous flow apparatus to separate large quantities of proteins is desired. An early phase of this effort identified drug companies that need improved separation, and it was shown how the limitations of ground separation could be overcome in space. Various companies have supplied protein samples to evaluate overall limitations of the system on Earth and have expressed interest in continuing the interaction. A continuous flow electrophoresis chamber for improved separation on the ground and in space has been built and protein samples from various pharmaceutical companies have been separated. To demonstrate the effect of sample concentration on resolution, proteins of various densities will be used. Deflections will be measured, and the sample concentration distribution in buffer which is compatible with the protein's specific gravities will be measured. Model protein samples will be resolved to establish optimum separation conditions that compare with other systems. Operation of the 60 cm and 100 cm chamber will be compared for resolution, throughput dispersions of sample, optimum operational conditions, etc. Applied power and buffer flow rate will be adjusted to give best comparison.

### Publications

Weiss, R. A., Lanham, J. W., et al., "Investigation of the Free-Flow Electrophoresis Process," Final Report, NAS8-32200, May 1979.

## Electrophoresis Technology

Marshall Space Flight Center

Dr. R. S. Snyder

In-House      \$60K/year

The objectives of this program are to: (1) analyze the fluid flow and particle motions during continuous flow electrophoresis by experimentation and computation, (2) characterize and optimize existing electrophoretic separators and their operational parameters, and (3) separate biological cells using apparatus that has been characterized or modified to perform in a predictable manner and according to procedures that have been developed to yield improved separations.

The following results have been accomplished: (1) experiments have been designed to decouple or minimize the fluid effects due to the flow process, electrokinetic effects, and temperature gradients; (2) transparent electrophoresis chambers have been built allowing measurement of internal and wall temperature while observing flow perturbations; (3) techniques have been developed to map the temperature and flow fields in the chamber with small disturbance to the process; (4) a model has yielded results that reproduce flow distortions observed in experimental chambers; (5) the DESAGA F48 and Beckman continuous flow electrophoresis chambers have been compared with standard particles (fixed red blood cells) under various operating conditions. Optimum operating parameters for resolution and throughput have been established and the two devices can be compared; and (6) these optimized conditions are being used for the separation of biological cells with reproducibility.

### Publications

Rhodes, P. H., "Sample Stream Distortion Modeled in Continuous Flow Electrophoresis," NASA TM-78178, August 1979.

Snyder, R. S. and McGuire, J. K., "Characterization of Continuous Flow Electrophoresis for Improving Resolution and Throughput," in preparation.

## Mathematical Models of Continuous Flow Electrophoresis

Princeton University

Dr. D. A. Saville

Dr. R. S. Snyder, MSFC

NAS8-32614 Total Cost: \$178K (approx.)

November 1, 1977 - June 10, 1980

Development of high-resolution continuous flow electrophoresis devices ultimately requires comprehensive understanding of the ways various phenomena and processes facilitate or hinder separation. A comprehensive model of the actual three-dimensional flow, temperature and electric fields shall be developed to provide guidance in the design of electrophoresis chambers for specific tasks and means of interpreting test data on a given chamber.

Part of the process of model development shall include experimental and theoretical studies of hydrodynamic stability. This is necessary to understand the origin of mixing flows observed with wide-gap gravitational effects; the suppression of gravity may allow other processes to become important.

To insure that the model accurately reflects the flow field, particle motion requires extensive experimental work. Much of the experimental work can be done under terrestrial conditions if the roles of gravity are appreciated and taken into account properly. Even though the resolution of a terrestrial-based machine may be unsatisfactory, verification of the model will provide the support necessary for the interpretation of microgravity operations. Recommendations shall be made for the design and operations of the ground experiments.

### Publications

Saville, D. A., "Fluid Mechanics and Electrophoresis," Physiochemical Hydrodynamics, Guernsey: Advance Publications, Vol. 2, 1978, pp. 893-912.

Saville, D. A., "Fluid Mechanics and Continuous Flow Electrophoresis," COSPAR Space Research, Proceedings of XXI COSPAR Conference, Innsbruck, Austria, Vol. XIX, Pergamon Press, 1979, pp. 583-597.

Saville, D. A., "Flow Structure and Stability in Continuous Flow Electrophoresis," accepted for publication in Physicochemical Hydrodynamics.

Saville, D. A., "Studies of Continuous Flow Electrophoresis," AIAA Paper 79-0032, New York: American Institute of Aeronautics and Astronautics, 1979, 4 pgs.

**E. ULTRAHIGH VACUUM AND CONTAINERLESS  
PROCESSING TECHNOLOGIES**

## Ultrapure Metals Preparation in Space

University of California - Los Angeles  
Dr. R. F. Bunshah  
Dr. W. A. Oran, MSFC  
NAS8-33115      \$67K/year  
August 7, 1978 - March 7, 1980

The objective of this program is to investigate the preparation of ultrapure metals in space by vacuum melting/distillation technique and to study the properties of ultrapurity metals (impurity content  $< 1$  ppb).

The ultrapure high vacuum environment of an orbiting Molecular Wake Shield Facility or equivalent represents a unique environment for the preparation of ultrapure metals because it provides a very low pressure environment of contamination gas. Starting with vacuum-melted material, high-purity samples of chromium, vanadium, and molybdenum were prepared in a diffusion pump system by vacuum distillation at three different base pressures in the range of  $10^{-4}$  and  $10^6$  Torr for each metal. Samples were analyzed for various metallic and nonmetallic impurity content. Decreasing pressure of the vacuum system resulted in lower non-metallic impurity content, as might be expected. Best results were observed for vanadium. While there were few discrepancies in the chemical analysis of metallic elements, the analysis showed that the impurity levels of these elements also decreased as the base pressure decreased.

### Publications

Agarwal, P., Bunshah, R. F., and Bevolo, A. L., "Ultrapure Metals Preparation in Space," Final Report, NAS8-33115, July 1980.

## Electrotransport of Solutes in Refractory Metals

Ames Laboratory, Iowa State University

F. A. Schmidt

H-34328B \$20K/year

October 1, 1978 - December 14, 1979

The objective of this investigation was to further study electrotransport behavior of metallic solutes in refractory metals. This work complemented a previous project in which a prototype electrotransport experiment was developed for deployment in a molecular wake shield facility. Specific experiments were made in which the electric mobility, diffusivity, and effective valence of molybdenum, rhenium, tungsten and zirconium in thorium were determined in an effort to better understand the behavior of these solutes so that maximum efficiency of the electrotransport refining process can be achieved.

A space simulation chamber capable of achieving pressures of  $2 \times 10^{-12}$  Torr was designed, fabricated, and used to develop a prototype electrotransport experiment. Laboratory electronics for the experiment were also developed, and an automatic control system was used to heat the specimen. Complete assembly line drawings of the components of the experiment and specifications for a high-current power supply were developed. Composite rods of thorium metal having a sharp step in the concentration of the solutes of interest were resistively heated to various temperatures with a direct current in a low-density environment. The displacement of this interface was traced by laser ion mass spectrometry from which the velocity of transport, electric mobility, diffusivity and effective valence of the solutes were determined. All four solutes were found to migrate in the same direction as the electron flow. Rhenium and molybdenum were found to be very mobile, with tungsten somewhat slower. Zirconium was found to move at a rate near that of the self-diffusion of thorium, viz., approximately two orders of magnitude slower than rhenium and tungsten.

### Publications

Lunde, B. K. and Schmidt, F. A., "Light Sensor for Ultra-high Vacuum System Utilizing a Silicon Photovoltaic Cell," Appl. Optics, 15, 1976, p. 2625.

Lunde, B. K. and Schmidt, F. A., "Precision Sample-and-Hold Stores Voltage for a Week," Electronic, Vol. 49, 1976, p. 120.

Scherber, F. A., Schmidt, F. A., et al., "Pressure Dependence of the Fermi Surface of Th," Phys. Rev. B, Vol. 16, 1977, p. 4235.

Schmidt, F. A., Lunde, B. K., et al., "The Ultra Purification of Metals by Electrotransport Under Outer Space Environmental Conditions," Final Report, January 1977.

Schmidt, F. A., Outlaw, R. A., et al., "Electrotransport Purification of Thorium Under Low Pressure Conditions," J. Electrochem. Soc., Vol. 126, 1979, p. 1811.

Schmidt, F. A., Carlson, O. N., et al., "Electrotransport of Solutes in Refractory Metals," Final Report, January 1980.

Schmidt, F. A., Beck, M. S., et al., "Molecular Wake-Shield Precursor Experiment," Final Report, January 1979.



## UltraHigh-Vacuum Semiconductor Thin-Film Technology

Jet Propulsion Laboratory

Dr. F. J. Grunthaner

Dr. B. F. Lewis

Dr. J. Maserjian

In-Center \$80K/year (approx.)

July 7, 1978 - January 31, 1980

The objective of this program is to investigate ultrahigh-vacuum (UHV) semiconductor thin-film technology. The ultimate aim is the development of procedures for the production of cost-effective thin-film semiconductor solar cells. In order to achieve this objective, the process requirements and limitations of this UHV technology must be carefully examined in ground-based experiments.

The approach to semiconductor thin-film deposition emphasizes structural measurement at all stages of deposition or synthesis. It is essential to characterize all stages of growth and processing utilizing a full range of surface spectroscopies. Growth and analysis chambers must have a UHV interconnection to permit in situ measurements.

The deposition approach also emphasizes the synthesis of interfaces rather than simple material deposition. Growth by use of molecular beams in a UHV environment allows compositional control on a monolayer or even submonolayer scale. A monolayer or less of specific chemical species (or dopants) can have a profound effect on interface electronic properties. The desired abrupt compositional changes prohibit the use of high sample temperatures during or after deposition. Instead, nonthermal energy deposition by infrared, ultraviolet, and/or electron irradiation can induce desired chemical reactions and increased mobility at the growing surface.

The new techniques of in situ post-deposition laser annealing and grapho-epitaxial substrates have already been shown to produce excellent crystallization of amorphous films without degrading their compositional profiles.

## Ultravacuum Vapor Epitaxial Growth of Silicon

General Electric Company

Dr. C. A. Neugebauer

NAS8-33121 Total Cost: \$105K (approx.)

August 30, 1978 - March 31, 1980

The objective of this program is to develop an ultra-high vacuum deposition process for the growth of polycrystals of silicon onto a polycrystalline oriented metal substrate. An important objective is to establish vacuum and vacuum purity requirements for the process and possible rationale for extension of these experiments using the greatly improved vacuum conditions and pumping speeds available in a Space Vacuum Research Facility. In addition, experiment requirements for possible extension of the vacuum deposition work in a Space Vacuum Research Facility are to be documented. Support of the system definition design work for such a facility is to be provided.

Silicon films 5 to 10 microns thick were deposited onto 25 to 50 micron tungsten foils from a resistively heated silicon source. Vacuum levels in the low  $10^{-8}$  Pa range and substrate temperatures in the 450°C to 600°C range were employed. X-ray diffractometry, scanning and transmission electron microscopy, and metallographic examination of cross-sectioned and etched films were among the methods used to examine the deposited films.

It was concluded that oriented deposits can be obtained at substrate temperatures below 600°C. Two types of Si crystallites were distinguished: subgrains of 1 micron or less diameter which were columnar and only slightly misaligned with respect to each other, and large parent grains subtending many subgrains and coinciding with the tungsten grains in the substrate. Increasing the gas pressure leads to more random deposits, indicating lower pressure might lead to more ordered growth.

### Publications

Frost, R. T., et al., "Ultra Vacuum Epitaxial Growth of Silicon," to be presented at 8th International Vacuum Congress, Cannes, France, September 22-26, 1980.

## Efficient Solar Cells by Space Processing

Ames Laboratory, Iowa State University

F. A. Schmidt

J. R. Williams, MSFC

H-34328B      \$80K/year

October 1, 1976 - December 14, 1979

The purpose of this work was to define a laboratory experiment that demonstrates the feasibility of preparing efficient Schottky barrier solar cells by space processing. The investigation was directed toward utilizing the non-contaminating space environment in the preparation of thin film silicon solar cells by epitaxial growth on various metal substrates. The resulting deposits were then characterized to determine which parameters are essential to enhance solar cell efficiency.

Thin films of electron beam evaporated silicon were deposited on molybdenum, tantalum, tungsten and molybdenum disilicide under ultrahigh vacuum conditions. Mass spectra from a quadrupole residual gas analyzer were used to determine the partial pressure of 13 residual gases during each processing step. Surface contamination and interdiffusion were monitored by in situ Auger electron spectrometry. The study produced three important results, namely: (1) metal silicide growth can be suppressed by use of a silicon source heavily doped with phosphorous, (2) the partial pressure of oxygen is influential upon the silicon grain size even at the  $10^{-10}$  Torr level, and (3) the first known preparation of silicon heteroepitaxy on a metallic substrate, namely  $\text{MoSi}_2$ , was achieved.

### Publications

Schmidt, F. A., Campisi, G. J., et al., "Efficient Solar Cells by Space Processing," Final Report, January 1980.

Schmidt, F. A., Shanks, H. R., et al., "The Preparation of Thin Film Solar Cells Under Very Low Pressure Conditions," Final Report, December 1977.

Bevolo, A. J., Schmidt, F. A., et al., "Polycrystalline Silicon on Tungsten Substrates," J. Vac. Sci. Technol., 16, 1979, p. 13.

Crystal Nucleation in Glass-Forming Alloy and Pure Metal  
Melts Under Containerless and Vibrationless Conditions

Harvard University

Professor David Turnbull

NAS8-32691 Total Cost: \$120K (approx.)

June 23, 1978 - June 22, 1981

The main objective of this research is to characterize nucleation behavior in glass-forming alloy melts. Such experiments should indicate if formation of alloy glasses in bulk form is possible and, if so, what are the necessary conditions. The most favorable conditions would be those in containerless, vibrationless experiments in high vacua or inert atmospheres.

The crystal nucleation behavior of  $\text{Au}_4\text{Si}$  and  $\text{Pd}_4\text{Si}$  glass-forming melts and of pure Ni by the droplet technique is being investigated. It has been found that the onset undercooling,  $T_0$ , for copious nucleation in molten  $\text{Au}_4\text{Si}$  droplets varies widely with thermal treatments which alter the nature of the  $\text{SiO}_2$  film on the droplet surface. However,  $T_0$  as large as 1/3 of the liquidus temperature for some droplets was observed. Glass and crystallization temperatures of  $\text{Au}_4\text{Si}$ -based alloys are sharply increased ( $\sim 1^\circ$  per atom %) when Cu replaces some of the Au.

Publications

Turnbull, D., "On Anomalous Prefactors from Analysis of Nucleation Rates," NASA Quarterly Report, September 1979.

Spaepen, F. and Turnbull, D., "Negative Pressures and Melting Point Depression in Oxide-Coated Liquid Metal Droplets," Scripta Met., Vol. 13, 1979, p. 149.

Thompson, C. V. and Spaepen, F., "On the Approximation of the Free Energy Change on Crystallization," Acta Met., Vol. 27, 1979, p. 1855.

A Proposal to Measure the Viscosity of Molten Pd<sub>78</sub>Si<sub>16</sub>Cu<sub>6</sub>  
A Critical Experiment to Support Possible Space Flights

Drexel University  
Dr. Arthur E. Lord  
NAS8-3372 \$29K/year (approx.)  
February 1979 - June 1980

Metallic glasses are materials that are cooled very rapidly to bypass crystallization. Due to the lack of crystallinity, these materials have very unique properties (very "soft" magnetic properties, high hardness, high yield stress, corrosion resistance, etc.) In general, only small sizes are available. To try and make larger samples the time-temperature transformation (TTT) curves must be determined in order to calculate the cooling rate ( $R_c$ ) needed for glass formation. The most important physical property needed for the TTT diagram calculation is the viscosity and its temperature dependence.

An oscillating crucible method for determining the viscosity of molten Pd<sub>78</sub>Si<sub>16</sub>Cu<sub>6</sub> alloy has been developed. This material is one of the easiest glass formers and hence can be made in bulk. The results for the auxiliary eutectic alloy Pd<sub>82</sub>Si<sub>18</sub> indicate an extremely high viscosity about 50 poise at the melting point. This is about 1000 times the value for other liquid metal systems. The viscosity of Pd<sub>78</sub>Si<sub>16</sub>Cu<sub>6</sub> is presently being determined, and with these data the calculations for the best TTT diagram viscosity will be obtained. Interesting results have also been obtained for the viscosity of Fe-Ni-P-B alloys.

Publications

Steinberg, J. and Lord, A. E., "The Viscosity of Molten Fe-Ni-P-B Alloys," J. Noncryst. Solids (submitted for publication).

Steinberg, J. and Lord, A. E., "The Viscosity of Molten Pd<sub>82</sub>Si<sub>18</sub> Alloy," J. Noncryst. Solids (submitted for publication).

Lord, A. E., Wouch, G., and Frost, R. T., "Preparation of Amorphous Ferromagnetic Materials through Containerless Solidification," SPAR IV Report, NASA TM-78235, January 1980.

## Fusion Target Technology

Jet Propulsion Laboratory

Dr. T. G. Wang

In-Center \$300/year

October 1979 - continuing task

The objectives of this task are to (1) study the physical processes that are associated with the fabrication of inertial confinement fusion (ICF) targets in a weightless environment, (2) determine jointly with DOE centers the need for extended zero gravity in the future production of ICF targets, and (3) provide technological information to DOE centers that is pertinent to their current target fabrication research.

To produce the high-quality fusion target shells that are required, four fundamental physical processes must be understood: spheroidization of the shell, uniformity of shell thickness and coating, adiabatic expansion, and contraction of the molten pellet as it passes through temperature gradient environments.

The approach to be taken is to (1) study the fluid dynamic processes that pertain to pellet fabrication processes such as bubble centering, coating uniformity, and various instabilities; (2) study the effects of various temperature levels and temperature gradients on pellet fabrication; (3) construct Earth-based high temperature and high temperature gradient drop towers; (4) initiate development of a process for the studies, numerical analyses, and computer calculations on various pellet fabrication processes.

### Publications

Wang, T. S., "Gravitational Effects on Target Fabrication," Technical Digest of the Conference on Inertial Confinement Fusion, San Diego, California, February 1980.

Elleman, D. D., Croonquist, A. P., et al., "Low Gravity; Acoustic Positioning Chamber for Liquid Shell Studies," Technical Digest of the Conference on Inertial Confinement Fusion, San Diego, California, February 1980.

Lee, M. C., Helizon, R. S., et al., "Real-Time Image Acquisition and On-line Image Processing of a Hollow Sphere Confined in a Drop Furnace," Technical Digest of the Conference on Inertial Confinement Fusion, San Diego, California, February 1980.

Kendall, J. M., "Fluid Dynamics Relating to the Production of Large Fusion Targets," Technical Digest of the Conference on Inertial Confinement Fusion, San Diego, California, February 1980.

## The Upgrading of Glass Microballoons

Bjorksten Research Laboratories

Dr. Stanley A. Dunn

Robert T. Nagler

Elmer G. Paquette

Stephen Gunter

Dr. E. J. Crosby

NAS8-33513 \$43.8K/year (approx.)

August 28, 1978 (repropose each 12 months)

The objective of this program is to study extensively the processes and mechanisms involved in producing glass microballoons of acceptable quality for laser fusion by gas jet levitation and manipulation in the molten condition.

The gas supporting levitation microfurnace (LMF) concept represents a unique means of handling and shaping small bodies where contact with foreign condensed phase objects or supports could be deleterious. Levitation is achieved by aerodynamic lift imparted to the body from a gaseous flow field generated with the aid of a collimated hole structure (CHS). A long-standing objective of the development of the LMF has been the perfecting of glass microballoon (GMB) geometry by centrifugal centering and coordinated temperature and pressure manipulation. Centering by centrifugal means implies the ability to control the orientation of the axis of rotation as well as the speed.

Existing LMF designs provide excellent horizontal and vertical translational stability. A third requirement, that of rotational control has been partially solved. Rate of rotation can be controlled down to very slow speeds. The ability to stop rotation entirely and to index the GMB to any desired orientation are objectives now being sought through division of the field of jets into three or four regions, each with separately controllable gas flows. The capability of being able to stop rotation as well as to control position will be of use in studies of heat transfer involving GMB's.

### Publications

Dunn, S. A. and Gunter, S., "The Upgrading of Glass Microballoons - Targets for Laser Fusion," NASA CR-161280, 1979.



## Glass Shell Manufacturing in Space

KMS Fusion, Inc.  
Dr. Robert L. Nolen  
NAS8-33103      Total Cost: \$266K (approx.)  
December 6, 1978 - December 5, 1981

The principal objective of this research is to develop a detailed understanding of the chemical and physical processes involved in the formation of uniform, high-quality spherical glass shells. Where possible, various stages of the shell-blowing process will be formulated into mathematical models.

The temperature regimes where major transformations occur in the shell starting materials (metal-organic gels) have been identified. This was accomplished using a combination of thermal analytical techniques. The gases generated by pyrolysis of the gel were quantitatively characterized by gas chromatography and pressure tests.

A mathematical model of the heat transfer mechanisms occurring in the gel-to-shell transformation will be refined from one previously formulated under another contract. Computerization of the mathematical model will be used to bring it into line with the results from a series of controlled drop tower furnace experiments to be done with fully characterized and standardized gel powder pellets. Experiments conducted in the course of modeling the heat transfer process should also provide information on gas transport mechanisms.

### Publications

Nolen, R. L., "Glass Shell Manufacturing in Space," First-Fourth Quarterly Reports, NAS8-33103, March 1979-January 1980.

A Thermochemical Study of Corrosive Reactions in Oxide Materials

National Bureau of Standards  
Dr. H. S. Parker  
H-27354B \$80K/year (approx.)  
April 1977 - April 1980

The objectives of this task are to investigate the nature, extent and limitations on experimental studies imposed by sample container reaction at high temperature and to evaluate containerless techniques for melt and solid state investigations.

The general approach taken was to select a system of current technological importance and investigate: (a) maximum temperature limits for the container materials and characterize the reaction products, (b) determine the equilibrium phase assemblages in selected portions of the system within the limitations imposed by (a), and (c) in collaboration with MSFC provide specimens for levitation experiments at MSFC and correlate the results of levitation experiments with container experiments to evaluate problems associated with low gravity experiments.

The system being investigated was the  $K_2O$ -iron oxide- $SiO_2$  system. This system is of importance to the MHD effort, where these oxides are the major components of seed and coal slag. The first portion investigated consisted of compositions on the  $KFeO_2$ - $Fe_2O_3$  binary. Because of the high temperatures involved ( $T > 1500^\circ C$ ), platinum was chosen as the container material. As a result of this study, it was discovered that, contrary to the expected result, platinum was a suitable container, provided most of the Fe was kept in the 3+ state. Also, the existence of an alumina structure-type phase was reported.

Publications

Parker, H. S., Roth, R. S., et al., "NBS: Properties of Electronic Materials," NBSIR 78-1483, edited by J. R. Manning and R. L. Parker, 1978, pp. 84-100.

Parker, H. S., Roth, R. S., et al., "NBS: Properties of Electronic Materials," NBSIR 79-1767, edited by J. R. Manning, 1979, pp. 82-102.

Measurement of the Properties of Tungsten at High  
Temperatures

Rice University  
Dr. J. Margrave  
NAS8-33199     \$20K/year  
November 27, 1978 - June 1, 1980

This research is directed toward the measurement of the thermophysical properties of tungsten using containerless techniques. The properties of tungsten are of interest because it lies at the extreme of metal melting points and is a key datum in any extrapolation or interpolation process. In addition, difficulties in handling molten tungsten may establish the limitations of ground-based containerless systems in processing materials at high temperatures.

Samples are suspended containerlessly by an electromagnetic levitator. Additional heat is supplied by electron bombardment. Temperatures are measured by pyrometers. Heat capacities are determined from cooling curves, and total enthalpy is obtained using a drop calorimeter.

### Ultimate Intrinsic Coercivity SmCo<sub>5</sub> Magnet

Charles Stark Draper Laboratory  
Dr. Dilip K. Das  
Dr. R. T. Frost, General Electric  
NAS8-33607      Total Cost: \$396K (approx.)  
September 24, 1979 - September 23, 1982

The objective of the proposed research is to produce Sm-Co magnets of reasonably high maximum energy product with intrinsic coercivity approaching the theoretical limit of 350 KOe. These magnets will have to be composed of extremely fine, defect-free crystallites. The goals will be accomplished by (1) preparation of highly pure Sm-Co alloy by levitation melting and solidification in oxygen-free vacuum, (2) comminution of the alloys to micron size particles in oxygen-free environment, (3) densification of the aligned alloy powder to near theoretical density without introducing contamination and preventing grain growth.

Even though permanent magnets fabricated from Sm-Co are the strongest known, their coercivities are in the neighborhood of only 30 KOe, a mere 10 percent of the theoretical potential. The reason for this low value has been determined to be the reversal of magnetization by the mechanism of domain nucleation (at comparatively small demagnetizing fields at defect sites) and motion of domain walls.

The main task will be to develop alloy comminution techniques for the production of fine powders in an oxygen-free environment and densification of the magnetically aligned powders to near theoretical density in oxygen and other contamination-free states with fine grain size.

## Undercooling Studies in Metastable Peritectic Compounds

Marshall Space Flight Center  
Dr. L. L. Lacy  
In-House \$80K/year  
March 1, 1979 - March 1, 1982

The objective of this program task is to investigate undercooling and containerless solidification of metastable superconducting alloys  $Nb_3Ge$  and  $Nb_3Al$  and pure metal melts such as Nb; specifically, to investigate the structure and superconducting properties of undercooled Nb-Ge alloys and to determine the feasibility of forming metastable  $Nb_3Ge$  in bulk form.

Pure Nb droplets have been undercooled in excess of 500 K in free fall using the MSFC drop tube. The droplets form single crystals with no shrink cavity in the interior. The outer surface is rough, indicating the shrinkage associated with solidification was taken up by the interdendritic fluid.  $Nb_3Ge$  droplets have also been deeply undercooled and rapidly solidified in the drop tube. Unfortunately, accurate measurements of the amount of undercooling were not available at the time. An increase in transition temperature of  $\approx 2$  K was observed above the normal as-cast transition temperature (usually about 6 K), indicating that some of the A-15 structure may have been formed. New instrumentation has been added to the drop tube to obtain an accurate temperature history of the sample. The tests with the  $Nb_3Ge$  droplets will be repeated so that the superconducting performance can be related to the amount of undercooling.

### Publications

Lacy, L. L., Robinson, M. B., and Rathz, T., "Drop Tube Experiments on Undercooled Niobium and Niobium Alloy Droplets," 108th AIME, New Orleans, 1979.

Lacy, L. L., Robinson, M. B., "Containerless Undercooling and Solidification in Drop Tubes," submitted to J. Colloid and Interface Science.

Robinson, M. B., Rathz, T. J., et al., "Drop Tube Apparatus for Low-Gravity Solidification," NASA Tech Briefs, MFS-25242, 1980 (patent pending).

Nisen, N. B., Lacy, L. L., and Robinson, M. B., "Containerless High-Temperature Calorimeter," NASA Tech Briefs, Spring 1979, p. 89 (patent pending).

Steinberg, J., Lord, A. E., Lacy, L. L., et al., "Production of Bulk Amorphous  $\text{Pd}_{77.5}\text{Si}_{16.5}\text{Cu}_6$  in a Continuous Low-Gravity Environment, accepted for publication in Applied Phys. Letters.

Rathz, T. R., "Superconducting Measurements on Nb-Ge Alloys Using Susceptibility Techniques," MS Thesis, University of Alabama in Huntsville, 1980.

## Containerless Processing Technology

NASA Marshall Space Flight Center

Dr. W. A. Oran

L. H. Berge

B. B. Ross

Dr. J. E. Rush, UAH

In-House Cost: N/A

The objective of this program is to develop the research area of containerless processing in a microgravity environment, including: (1) the organization of working groups of experts and potential users to help develop a ground-based research program and define possible flight hardware, (2) the development and, in part, definition of the limitations of various ground-based containerless processing techniques, and (3) the conduction of precursory experiments in these ground-based assemblies.

Single-axis acoustic levitation devices were utilized as a basis for developing a system to containerlessly process nonconducting materials. However, there are apparent difficulties in operating one of these devices in a ground-based processing system. If one attempts to spot heat a levitated specimen to a high temperature in a relatively cold gaseous medium, instabilities are apparently established in the acoustic standing wave field which result in the sample being ejected from the levitation well. Acoustic streaming influences the temperature of a specimen levitated in an "isothermal" furnace. The streaming pumps cooler air into the levitation region in a manner such that the temperature of the specimen appears influenced more by the transducer temperature than by the wall temperature.

A study of the use of airjet levitation devices to process nonconducting materials has begun. Airjet systems have been used to containerlessly process metals, although the EM generator was used for heating and the resulting field greatly helped to stabilize the metal when molten.

### Publications

Oran, W. A., "Proceedings of the Workshop on an Electromagnetic Positioning System in Space," NASA CP-2069, October 1978.

Oran, W. A., "Preliminary Characterization of a One-Axis Acoustic System," NASA TM-78213, January 1979.

Oran, W. A., "A Parametric Study of an Acoustic Levitation System," Rev. Sci. Instrum. 51, 626 (1980).

Oran, W. A., "Some Limitations in Processing Materials in Acoustic Levitation Devices," IEEE Transactions on Ultrasonics, September 1979, pp. 482-486.

Oran, W. A., "Containerless Melting and Solidification," Proceedings of Electrochemical Society Meeting, St. Louis, May 1980.

Oran, W. A., "Containerless Processing of Metals with an Electromagnetic Levitation System," in preparation.



Thermodynamic Properties of Refractory Inorganic Materials  
at High Temperatures

National Bureau of Standards

J. H. Colwell

H-27954B \$50K/year (approx.)

April 1978 - continuing task

The objective of this research is to develop containerless techniques for determining thermophysical properties of nonmetallic samples at high temperatures.

Two approaches have been considered for carrying out the low-gravity experiments. The first is analogous to a drop-calorimetry experiment in that the sample would be heated to a predetermined temperature whereupon its enthalpy would be determined by engulfing the sample in a calorimeter. This approach would be tedious since a single point would be obtained for each sample, and many points would be needed for the requisite data. The second approach that is presently being pursued involves heating a sample to a high temperature and then allowing it to freely cool while simultaneously measuring its surface temperature and total radiant energy loss as a function of time. Temperature gradients within the samples will complicate the interpretation of the experiments but will also permit thermal conductivities of the samples to be determined under certain conditions. The limiting parameters for these measurements are being determined by computer modelling experiments. Pyroelectric detectors are being tested for potential use in the measurement of the total radiation from the samples.

Publications

Colwell, J. H., "NBS: Properties of Electronic Materials," NBSIR 79-1767, edited by J. R. Manning, 1979, pp. 103-131.

## Advanced Containerless Processing Technology

Jet Propulsion Laboratory

Dr. T. G. Wang

In-Center \$350K/year

October 1978 - continuing task

The primary long-range objectives of this task are to: (1) study and advance the science of contactless positioning and manipulation of a high-temperature acoustic chamber, (2) provide design information on a flight version of this chamber for materials science studies in a contactless and zero gravitation environment, and (3) provide potential MPS investigators with a set of ground-based facilities with which to perform precursor experiments.

The approaches to be taken in FY 81 are experimental and theoretical studies of (1) acoustic positioning and manipulation capabilities of a rectangular chamber as a function of temperature and pressure, (2) various acoustical geometries which may have special application in materials science studies, (3) loss mechanisms associated with high-intensity and high-temperature acoustic waves, (4) an aeroacoustic positioning system which will allow us to levitate heavy samples in the laboratory, (5) a liquid-liquid positioning system which will allow us to study the dynamic of liquid melts, and (6) positioning and manipulation capabilities of a KC-135 acoustic module.

In addition, a new effort will be initiated this year to provide MPS investigators with a set of facilities to perform precursor experiments prior to spaceflight. These facilities will allow investigators to examine and compare the properties of their sample processes in the following four ways: one-g contained, one-g containerless, zero-g contained, and short-duration zero-g containerless.

### Publications

Lagomarsini, G. and Wang, T. G., "Acoustic Containerless Processing Module for Materials Research," AIAA, 79-0369, 1979.

Jacobi, N., Tagg, R. P., et al., "Free Oscillations of a Large Drop in Space." AIAA, 79-0225, 1979.

Lee, M. C., "Temperature Dependence of Molecular Vibrational Relaxation Frequency for  $O_2-H_2$ ,  $O_2-He$  and  $O_2-CO_2$  Mixture Between 300-675°K," Proceedings of the Ultrasonics Symposium, IEEE, New Orleans, September 1979.

Barmatz, M. B., "Nonlinear Effects in a High Intensity Acoustic Resonant Chamber," Proceedings of the 10th International Congress on Acoustics, Sydney, Australia, July 1980.

Trinh, E., Wang, T. G., and Lee, M. C., "A Technique for the Study of Drop Dynamics in a Liquid-Liquid System," JASA 67, S1 (1980).

Wang, T. G., "Acoustic Levitation and Manipulation for Space Applications," Proceedings of the Ultrasonics Symposium, IEEE, New Orleans, September 1979.

Wang, T. G., Elleman, D. D., et al., "Containerless Processing Technology Experiment Report, SPAR IV Experiment 76-20, SPAR IV Final Report, NASA TM-78235, January 1980.

Electrostatic Control and Manipulation of Materials for  
Containerless Processing

Jet Propulsion Laboratory

Dr. M. M. Saffren

Dr. D. D. Elleman

Dr. W. K. Rhim

In-Center \$250K (to date)

October 1978 - continuing task

The objective of this task is to develop electric field positioning/manipulation techniques and technology for the containerless processing of materials in bulk and dispersed forms. This method obviates limitations of other methods. Two principal demonstrations to prove technique and technology will be completed no later than CY 83. An important part of the task is to ensure that facilities to be developed satisfy requirements of identified user applications. Another part of the task is to pursue study of the electrohydrodynamics of liquid drops (that must be done as part of the development) as a fundamental investigation.

Task studies include: methods and limitations of electric field confinement, electrode configuration, electrohydrodynamics of both charged and neutral drop systems, and dynamics and stability of charged particle arrays. These investigations are performed in the laboratory, in the KC-135 aircraft, and through theoretical and numerical study. Use is made of available SPAR flight experiment data, and Shuttle flight experiments are planned.

Publications

Saffren, M. M., Elleman, D. D. and Rhim, W. K., "Dynamics of Liquid Shells," Conference on Inertial Confinement Fusion, San Diego, California, February 26-28, 1980.

Saffren, M. M., Elleman, D. D., and Rhim, W. K., "Dynamics of Compound Liquid Drops," JPL Report 715-27: "Theoretical Development--Concentric Drops--Mode Frequencies and Interfacial Displacements," Vol. I; "Theoretical Development--Energy and Angular Momentum," Vol. II; "Numerical Results," Vol. III; "Non-Concentric Modes and Centering Forces--Theoretical Development," Vol. IV.

## Mass Spectrometer Development

NASA Langley Research Center  
Dr. Leonard L. Melfi  
In-Center \$0K  
October 1979 - October 1980

The technical objective of this task is to design, develop, and fabricate the molecular-beam mass spectrometers required to measure the density of gas species in the vicinity of the Space Shuttle orbiter.

The mass spectrometer design has been based on drifting gas kinetic theory and molecular-beam techniques to guarantee that the instrument output is independent of gas surface interactions and outgassing. Ultraclean materials are used in the fabrication of each component of the mass spectrometer. Further, these components are fired at 1000°C at  $10^{-8}$  Torr to substantially reduce instrument outgassing. Ultraclean vacuum brazing and electron beam techniques are used to assemble the mass spectrometer. The mass spectrometer is a quadrupole which uses ion counting techniques. The ion source is an electron impact type which uses a low-temperature hot cathode as the electron source. The electronics are designed to provide adequate stability for long counting intervals.

### Publications

Melfi, L. T. and Brock, F. J., "Molecular Beam Techniques Applied to Mass Spectrometer Thermospheric Density Measurements," Rev. Sci. Instrum., Vol. 144, October 1973.

Melfi, L. T., Brock, F. J., et al., "A New Approach to Thermospheric Mass Spectrometer Density Measurements," NASA TN D-7711, September 1974.

Clay, F. P., Brock, F. J., et al., "A Switching Regulator Emission Control Circuit for Ion Sources," Rev. Sci. Instrum., Vol. 46, May 1975, pp. 528-532.

Clay, F. P., "Demonstration of Phase Relationships in a Series RLC Circuit Using a Four-Channel Multiplexer and a Single-Channel Oscilloscope," Am. J. Phys., Vol. 47, April 1979, pp. 327-340.

Clay, F. P., et al., "The Emission Efficiency of a Hot Filament in the Presence of  $O_2$  at Various Pressure and Techniques for Maintaining Constant Emission," Va. J. Sci. Vol 24, February 1973.

Melfi, L. T., Outlaw, R. A., et al., "Gold-Plating-Brazing of Ion Source Components," Va. J. Sci., Vol. 26, Summer 1975.

Clay, F. P., Melfi, L. T., et al., "Evaluation of a Thorium-Coated Iridium Cathode for an Ion Source," Va. J. Sci., Vol. 26, Summer 1975.

Technology Demonstration Measurement for Molecular  
Wake Shield

University of Texas - Dallas

Dr. J. H. Hoffman

R. R. Hodges

W. W. Wright

NAS8-32689 \$30K/year (approx.)

May 19, 1978 - June 1, 1980

This task has consisted of an investigation of concepts and techniques for measuring and characterizing the ultrahigh vacuum in the wake of an orbiting spacecraft. The means chosen to best satisfy the objectives of the investigation is employment of a dual mass analyzer concept, both units being magnetic sector mass spectrometers. One would contain a high-sensitivity, open ion source for neutral gas measurements; the other would be an ion mass analyzer, identical to the first but without an ion source. The environment is expected to consist principally of neutral and ionized light gases such as H, H<sub>2</sub>, He, O, O<sub>2</sub>, N<sub>2</sub>, and CO<sub>2</sub>.

Sensitivity of the neutral mass analyzer is 1 count/sec per 100 molecules/cm<sup>3</sup>, enabling the instrument to measure partial pressures in the range from 10<sup>-16</sup> Torr to 10<sup>-10</sup> Torr with integration times of the order of 30 sec. The ion analyzer will detect number densities from 0.1 to 10<sup>5</sup>/cm<sup>3</sup>. Mass ranges of the neutral and ion analyzers are from 1 to 100 and 1 to 64 amu, respectively, but can be easily be adjusted to the specific ranges desired for the wake analysis.

Publications

Hoffman, J. H., Hanson, W. B., et al., "The Magnetic Ion Mass Spectrometer on Atmospheric Explorer," Radio Science 8, 315-322, 1973.

Hoffman, J. H., "Ion Mass Spectrometer on Explorer 31 Satellite," Proc. IEEE, 57, 1063-1067, 1969.

Hoffman, J. H. and Hodges, R. R., "Molecular Gas Species in the Lunar Atmosphere," The Moon, 14, 159-167, 1975.

Yeager, P. R., Smith, A., et al., "Absolute Calibration of Apollo Lunar Orbital Mass Spectrometer," J. Vac. Sci. Technol., 10, 348-354, 1973.

System Feasibility of a Space Vacuum Research Facility -  
Wake Shield Demonstration (WSD)

McDonnell-Douglas Astronautics Company  
B. C. Moore  
NAS8-33155 Total Cost: \$55K  
16 months (includes 4 month extension)

The objective of this task was the definition of a wake shield demonstration (WSD) to prove the theory of ultrahigh vacuum behind the shield and the effectiveness of degassing and decontamination techniques.

Publications

Analysis of Degassing Techniques to Support Space Vacuum Research Facility, McDonnell-Douglas, NAS8-33155, Vols. IA, II, III, and V, September 11, 1979.

System Feasibility of a Space Vacuum Research Facility - Wake Shield Demonstration, McDonnell-Douglas, Vols. IB and IV, January 1980.



Analysis of Degassing Techniques to Support Vacuum Research Facility (SVRF)

McDonnell-Douglas Astronautics Company

B. C. Moore

NAS8-33155 Total Cost: \$99.9K

16 months

The objectives of this program task were: (1) to develop data on cleanliness levels and outgassing techniques, (2) document present aerospace technology and procedures in ultrahigh vacuum systems and high cleanliness spacecraft, (3) identify SVRF degassing methods, evaluate each method and select optimum, and (4) define scenarios for fabrication and operation of the SVRF.

Publications

Analysis of Degassing Techniques to Support Space Vacuum Research Facility, McDonnell-Douglas, NAS8-33155, Vols. IA, II, III, and V, September 11, 1979.

System Feasibility of a Space Vacuum Research Facility - Wake Shield Demonstration, McDonnell-Douglas, NAS8-33155, Vols. IB and IV, January 30, 1980.

## **F. BIOPROCESSING**

## Electrophoresis Technology

Marshall Space Flight Center  
Dr. R. S. Snyder  
In-House \$60K/year

The objectives of this program are to: (1) analyze the fluid flow and particle motions during continuous flow electrophoresis by experimentation and computation, (2) characterize and optimize existing electrophoretic separators and their operational parameters, and (3) separate biological cells using apparatus that has been characterized or modified to perform in a predictable manner and according to procedures that have been developed to yield improved separations.

The following results have been accomplished: (1) experiments have been designed to decouple or minimize the fluid effects due to the flow process, electrokinetic effects, and temperature gradients, (2) transparent electrophoresis chambers have been built allowing measurement of internal and wall temperature while observing flow perturbations, (3) techniques have been developed to map the temperature and flow fields in the chamber with small disturbance to the process, (4) analysis has yielded results that reproduce flow distortions observed in experimental chambers, (5) the DESAGA F48 and Beckman continuous flow electrophoresis chambers have been compared with standard particles (fixed red blood cells) under various operating conditions. Optimum operating parameters for resolution and throughput have been established and the two devices can be compared, and (6) these optimized conditions are being used for the separation of biological cells with reproducibility.

### Publications

Rhodes, P. H., "Sample Stream Distortion Modeled in Continuous Flow Electrophoresis," NASA TM-78178, August 1979.

Snyder, R. S. and McGuire, J. K., "Characterization of Continuous Flow Electrophoresis for Improving Resolution and Throughput," in preparation.

## Mathematical Models of Continuous Flow Electrophoresis

Princeton University

Dr. D. A. Saville

Dr. R. S. Snyder, MSFC

NAS8-32614 Total Cost: \$178K (approx.)

November 1, 1977 - June 10, 1980

Development of high-resolution continuous flow electrophoresis devices ultimately requires comprehensive understanding of the ways various phenomena and processes facilitate or hinder separation. A comprehensive model of the actual three-dimensional flow, temperature and electric fields shall be developed to provide guidance in the design of electrophoresis chambers for specific tasks and means of interpreting test data on a given chamber.

Part of the process of model development shall include experimental and theoretical studies of hydrodynamic stability. This is necessary to understand the origin of mixing flows observed with wide-gap gravitational effects; the suppression of gravity may allow other processes to become important.

To insure that the model accurately reflects the flow field, particle motion requires extensive experimental work. Much of the experimental work can be done under terrestrial conditions if the roles of gravity are appreciated and taken into account properly. Even though the resolution of a terrestrial-based machine may be unsatisfactory, verification of the model will provide the support necessary for the interpretation of microgravity operations. Recommendations shall be made for the design and operations of the ground experiments.

### Publications

Saville, D. A., "Fluid Mechanics and Electrophoresis," Physiochemical Hydrodynamics, Guernsey: Advance Publications, Vol. 2, 1978, pp. 893-912.

Saville, D. A., "Fluid Mechanics and Continuous Flow Electrophoresis," COSPAR Space Research, Proceedings of XXI COSPAR Conference, Innsbruck, Austria, Vol. XIX, Pergamon Press, 1979, pp. 583-597.

Saville, D. A., "Flow Structure and Stability in Continuous Flow Electrophoresis," accepted for publication in Physicochemical Hydrodynamics.

Saville, D. A., "Studies of Continuous Flow Electrophoresis," AIAA Paper 79-0032, New York: American Institute of Aeronautics and Astronautics, 1979, 4 pgs.

## Automated Analytical Electrophoresis Apparatus

University of Arizona - Tucson

Dr. Peter H. Bartels

NAS8-31948 Total Cost: \$300K (approx.)

June 4, 1976 - August 1980

The objectives of this program were to provide an automated apparatus to determine and display the mobilities of living cells suspended in a compatible buffer and to establish its capabilities as an instrument for research and clinical usage.

The rationale was to provide two types of data: histograms of mobilities and a statistical evaluation of the cell populations. Histograms may reveal a specific component of a cell population in a pathological specimen. Histograms also give prior notice of abnormal mobility distribution of the sample; this enables the search to be confined to abnormal subpopulations. Manual determination of electrophoretic mobilities is unsuitable for this purpose in that it lacks the ability to make a large number of precise measurements in a short period of time.

Research tasks included designing all the components and interfaces; building and testing a prototype to demonstrate design feasibility; commencing measurements of cell populations and monitoring apparatus performance parameters; and correcting deficiencies noted, including modifying apparatus as appropriate. The preceding tasks were completed to date. Measurements can be made faster than required. Limitations are photometric noise, accuracy of location determination and distance migrated at desirable voltages at select microscope magnification. Nevertheless, 1000 measurements per sample can be made within 10 minutes, with an optimum cell concentration of  $4 \times 10^6$  cells/cm<sup>3</sup> with 3 to 5 lock-ons per cell.

### Publications

Bartels, P. H., Bier, M., et al., "Automated Computer-Controlled Microscope Electrophoresis System," Cell Electrophoresis, April 1978, MIT, Cambridge, MA.

Brooks, D. E., et al., "Design and Testing of a Computer Controlled System (AEMS)," Cell Electrophoresis: Clinical Applications and Methodology, INSERM Symposium, Bristol, UK, 1979, pp. 409-419.

Bartels, P. H., et al., "Development Test and Evaluation of an Automated Analytical Electrophoresis Apparatus," Period May 17, 1976 - August 31, 1978, NASA/MSFC.

Bartels, P. H., et al., "Development Test and Evaluation of an Automated Analytical Electrophoresis Apparatus," Period September 1, 1977 - August 31, 1978, NASA/MSFC.

## Hormone Purification by Isoelectric Focusing in Space

University of Arizona - Tucson  
Dr. Milan Bier  
NAS8-32950 \$85K/year (approx.)  
March 29, 1978 - March 28, 1981

The objectives of this program are the development of ground-based prototype instrumentation for the purification of peptide hormones by means of isoelectric focusing; the development of accessory computer-controlled monitoring equipment for measurement of pH and ultraviolet adsorption; the development of a mathematical model of the focusing process; and an evaluation of the process for effect of gravity, leading to the definition of a possible space experiment.

Isoelectric focusing is generally recognized as having exceptionally high resolution for protein or peptide separation. The main shortcoming of previously available methods was that they were not applicable to large-scale preparative processes. To overcome this limitation, a new concept of recycling isoelectric focusing apparatus was proposed. The high capacity of this apparatus is due to its modular design and the recycling principle, which permit virtually limitless volumes to be processed, time being traded for volume capacity. A variety of biological materials have been processed, including synthetic ACTH and several glucagon derivatives, both being important peptide hormones, as well as several enzymes, proteins, and other peptide mixtures. Analysis of the effects of gravity on the process has shown that convective flows are present in adjacent compartments of the focusing apparatus differ in density, i.e., in solute content, but the extent of the harmful effects of this convection is not yet ascertained.

### Publications

Egen, N. B., Twitty, G. E., and Bier, M., "Recycling Isoelectric Focusing with Computer Controlled Data Acquisition System, 17th Aerospace Sciences Meeting, New Orleans, La, January 15, 1979, AIAA Paper 79-0405.

Bier, M. and Egen, N. B., "Large Scale Recycling Electro-focusing, in 'Electrofocuse/78', Hagland, Westerfield and Ball, Eds., Elsevier-North Holland, 1979.

Bier, M., et al., "New Developments in Isoelectric Focusing of Peptides," Proceedings of 6th American Peptide Symposium, in press.



## Countercurrent Distribution of Biological Cells

University of Oregon Health Sciences Center

Dr. D. E. Brooks

Dr. S. Levine

NAS8-33575      Total Cost: \$226K (approx.)

November 16, 1979 - November 15, 1982

The purpose of this program task is to develop a field-driven system for separating the polymer phases used in low-gravity phase partitioning experiments. Purification of biological cells by phase partitioning is impeded in unit gravity by the sedimentation of the sample material. This characteristic lowers the resolution capability of all phase partitioning separations, and disqualifies the method altogether for very large cells, some of which are of great biomedical interest. Moreover, fundamental investigations into the physiochemical mechanisms that underlie phase partitioning are themselves hampered by the influence of gravity.

Work begun aimed at providing phase diagrams for the dextran 500, dextran 400 and PEG 6 batches obtained for the contract work. These are necessary to define the effects of varying total polymer concentration on phase composition as well as to characterize the effects of added salts and changing temperature on the system. Polymer radiolabelling and polarimetry will be used in combination to provide the accurate and unambiguous phase composition values needed for making phase diagrams.

### Publications

Brooks, D. E., "SPAR Project: Countercurrent Distribution of Biologicals," Final Report, NAS8-32353, 1977.

Purification and Cultivation of Human Pituitary Growth  
Hormone Secreting Cells

Pennsylvania State University

Dr. W. C. Hymer

NAS9-15566 \$65K/year

June 1980 - June 1981

Human growth hormone (hGH) is in demand for the treatment of pituitary disease, osteoporosis, stress ulcers, etc. However, the demand is far greater than available supply. The problem could be solved by the culture of purified hGH cells that produce hormone. This program addresses the problem of (a) separation of the pituitary growth hormone cell, (b) its maintenance in vitro, and (c) assessment of the role that gravity plays in establishing limits of these current laboratory technologies.

In an effort to fulfill the above objectives a human pituitary column perfusion method has been developed to sustain hGH release from pituitary tissue over extended periods (1-3 days). On the basis of experimental results from 144 human pituitary glands removed 1-18 hours post-mortem, it has been found that prostaglandin E<sub>1</sub> ( $10^{-9}$  M) or epinephrine ( $10^{-9}$  M) stimulates release of a "GRF" from rat hypothalmi which is, in turn, capable of sustaining hGH release or at least 24 hours. Tissue samples stained immunocytochemically for hGH cells reveal large numbers of well-preserved cells in this experimental protocol. These results support the notion that the human post-mortem pituitary gland contains functional growth hormone cells.

Publications

Hymer, W. C., Page, R., et al., "Separated Somatotrophs: Their Use In Vitro and In Vivo, " Synthesis and Release of Adenohypophyseal Hormones (ed. Mckerns and Jutisz), Plenum Press, pp. 125-168, 1979.

Hymer, W. C., Wilbur, D. L., et al., "The Human Post Mortem Pituitary Contains Functional Somatotrophs," 62nd Meeting of the Endocrine Society, Washington, D.C., June 1980.

### Kidney Cell Electrophoresis

Pennsylvania State University  
Dr. Paul Todd  
NAS9-15584 \$40K/year  
June 1980 - continuing task

The objective of this investigation is to repeat and thereby validate the MA-011 experiment under laboratory conditions which are optimum for the viability of human kidney cells and most favorable for the best possible electrophoretic separation of those few (about 5%) cells which produce urokinase or human granulocyte conditioning factor (HGCF), and erythropoietin.

This study effort will perform the ground-based research necessary to establish all of the optimum experimental conditions required to accomplish the best possible electrophoretic separation of human kidney cell fractions, which produce urokinase, granulocyte stimulating factor, or erythropoietin. This overall effort will include: (1) development of optimum buffer systems, (2) viability tests, (3) ground-based research on electrophoretic mobilities, (4) development of standard cell culture methods and assays for urokinase, granulocyte stimulating factor, and erythropoietin, and (5) acquisition of the ground control data to be compared with results using cells returned from the electrophoretic separations carried out in microgravity.

It has been found in studies conducted on cells from 10 explants that electrophoretic heterogeneity exists in early-passage cultures and that cells from intermediate-mobility fractions appear to be most efficient in urokinase production. In tests of the flight buffer "A-1" it has been found that DMSO is superior to glycerol for cell freezing and that the low ionic strength required for electrophoresis is not a significant factor in determining cell viability.

## Electrophoretic Cell Separation Based on Immunomicrospheres

Jet Propulsion Laboratory  
Dr. Alan Rembaum  
Dr. Adam Smolka, UAB  
In-Center \$100K/year  
October 1978 - continuing task

Cell separation is needed for basic research as well as for diagnostic and chemotherapeutic purposes. The objective of this task is to demonstrate a new concept for the electrophoretic separation of immunologically labeled biological cells and to show that this separation is improved in space where sedimentation and convective flows due to gravity are minimized.

The electrophoretic mobility of human lymphocyte subpopulation (unlabeled T cells) was found to be about 40% higher than that of the labeled lymphocyte subpopulation (B cells). Experiments with sheep red blood cells were performed to evaluate the separation feasibility by means of the Hannig free flow electrophoretic instrument (DESAGA F48). Conditions for successful separation of unlabeled from labeled cells were determined. Preliminary runs with live lymphocytes on the DESAGA F48 yielded fractions with high concentrations of B cells recognized by intensely fluorescent immunomicrospheres attached to the cells as well as fractions with high concentration of non-fluorescent lymphocytes (presumably unlabeled T cells).

### Publications

Rembaum, A., "Microspheres as New Immunoreagents for Cell Identification," Flow Cytometry and Cell Sorting, Chapter 18, John Wiley & Sons, 1979.

Rembaum, A., Yen, S. P. S., et al., "Synthesis and Reactions of Hydrophilic Functional Microspheres for Immunological," J. Macrom. Sci. Chem. A., Vol. 13, p. 603.

Smolka, A. J. K., et al., "Electrophoretic Cell Separation by Means of Microspheres," Biochem. Biophys. Acta, Vol. 588, 1979.

Rembaum, A., "Synthesis Properties and Biomedical Applications of Hydrophilic Functional Polymeric Immunomicrospheres," Third International Colloid Conference, Stockholm, Sweden, August 1979.

Rembaum, A., "Electrophoretic Separation by Means of Immunomicrospheres," Australian-U.S.A. Seminar on Bioelectrochemistry, Pasadena, CA, July 10, 1979.

Biosynthesis/Separations Laboratory Development  
of a Space Biosynthesis System

Johnson Space Flight Center  
Dr. Dennis Morrison  
Dr. Bernard Mieszkuc  
In-Center     \$110K/year  
January 1980 - continuing task

The objectives of this program are to: (1) obtain data on the performance of cell culture vessel system elements and to define the biological oxidation process--the transfer of oxygen from gas to liquid and from liquid to oxidant, and (2) determine the limits of ground-based technology using a preprototype reactor for studying enzymatic reactions and suspension cell cultures.

The Biosynthesis/Separations Laboratory supports the Materials Processing in Space studies on biosynthesis and cell separations for investigations into the production of high value pharmaceuticals which are very difficult or impossible to obtain on Earth with currently available technology.

The laboratory has both monolayer and suspension cell culture capabilities. Current research includes procedures for the obtaining of cell cultures, the growth and maintenance of continuous cell cultures, and the freezing and storage of cells. Procedures for growing cell cultures in suspension are being investigated. A continuous line of baby hamster kidney cells has been grown in suspension, and the growth of cells on microcarriers is being pursued. A variety of beads were used as substrates for the attachment of cells. Procedures for the analysis of biochemicals produced by cell cultures have been established. Fibrinolytic and colorimetric methods are being used routinely for the assay of urokinase. The production of urokinase in monolayers of human embryonic kidney cells has been demonstrated. Biochemical purification of secreted products on affinity columns is being developed. Procedures for the chromosome analysis of cell cultures (counting and karyotyping) have been established. Follow-on efforts are expected to include the design, construction, verification testing, and flight test of a small space bioreactor to demonstrate the concepts and limitations of these new techniques using mammalian cells in culture which produce compounds of scientific or commercial importance.

**APPENDIX A**  
**MPS ORGANIZATIONS**

## MPS ORGANIZATIONS

Alabama A&M University  
Huntsville, AL

Arizona State University  
Tempe, AZ

Battelle Columbus Laboratories  
Columbus, OH

Bjorksten Research Laboratories  
Madison, WI

Case Western Reserve University  
Cleveland, OH

Charles Stark Draper Laboratory  
Cambridge, MA

Clarkson College of Technology  
Potsdam, NY

Drexel University  
Philadelphia, PA

EG&G Corporation  
Santa Barbara, CA

French Atomic Energy Commission  
Nuclear Research Center of Grenoble  
Grenoble Cedex, France

S. H. Gelles Associates  
Columbus, OH

General Electric Company  
Corporation Research Laboratories  
Schenectady, NY

General Electric Company  
Space Sciences Laboratories  
Valley Forge, PA

Grumman Aerospace Corporation  
Bethpage, NY



Harvard University  
Cambridge, MA

NASA  
Headquarters  
Washington, D. C.

Honeywell Research Laboratories  
Lexington, MA

Iowa State University  
Ames Laboratory, ERDA  
Ames, IA

Jet Propulsion Laboratory  
California Institute of Technology  
Pasadena, CA

NASA  
Johnson Space Center (JSC)

KMS Fusion, Inc.  
Ann Arbor, MI

NASA  
Langley Research Center (LaRC)  
Hampton, VA

Lehigh University  
Bethlehem, PA

A. D. Little, Inc.  
Cambridge, MA

Lockheed Corporation  
Huntsville Research & Engineering Center  
Huntsville, AL

Marvalaud Corporation  
Westminster, MD

Massachusetts Institute of Technology  
Cambridge, MA

NASA  
George C. Marshall Space Flight Center (MSFC)  
Marshall Space Flight Center, AL

McDonnell Douglas Corporation-West  
Huntington Beach, CA

McDonnell Douglas Corporation-East  
St. Louis, MO

National Bureau of Standards  
U. S. Department of Commerce  
Washington, D.C.

Northwestern University  
Evanston, IL

Pennsylvania State University  
University Park, PA

Princeton University  
Princeton, NJ

Rensselaer Polytechnic Institute (RPI)  
Troy, NY

Rice University  
Houston, TX

Rockwell International  
Downey, CA

Rockwell International  
Thousand Oaks, CA

Stanford University  
Stanford, CA

University of Alabama, Huntsville (UAH)

University of Arizona  
Tucson, AZ

University of California - Los Angeles

University of Oregon  
Health Sciences Center  
Portland, OR

University of Utah  
Salt Lake City, UT

**University of Wisconsin - Milwaukee**

**Westinghouse Corporation  
New Kensington, PA**

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APPROVAL

MATERIALS PROCESSING IN SPACE CATALOG OF TASKS, FY-80

Edited by R. J. Naumann

The information in this report has been reviewed for technical content. Review of any information concerning Department of Defense or nuclear energy activities or programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.



CHARLES A. LUNDQUIST  
Director  
Space Sciences Laboratory